CMC Bozeman Facility Supplemental Investigation Work Plan

Final Revision

Prepared for:

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1.0 BACKGROUND

On behalf of the City of Bozeman (City), Tetra Tech has prepared and is submitting this Final Revision Supplemental Investigation Work Plan (SI), for the CMC Bozeman Facility (Facility) property (Appendix A, Figure 1). This SI was promulgated due to a Department of Environmental (DEQ) requirement for additional investigation of specific areas in the Facility as documented to the City in a Proper and Expeditious Letter and Scope of Work (SoW) dated November 22, 2006. The SoW provides that information obtained through previous investigations may be utilized in the development of the SI work plan. Documents, such as the Voluntary Cleanup Plan for this Facility, containing information appropriate and relevant to this work plan will be cited herein and incorporated by reference. This SI work plan will address those components specified in the SoW, specifically, previously uninvestigated areas on property owned by Harrington's, Inc. where asbestos ore may be located, including unpaved areas; soils adjacent to the newer southern building addition on the south and east sides; the unpaved alley behind, and to the south, of Heeb's East Main Grocery (Heeb's); an evaluation of pavement condition of known/pavement-covered asbestos ore at the southwest corner of Wallace Avenue and Main Street; an evaluation of pavement condition of Wallace Avenue and sidewalks within the right-of-way running south from Main Street to Curtiss Street; investigation of the upper (northern) portion of the Story Distributing, Inc. property, and an investigation of potential airborne and settled dust concentrations of asbestos within the former ore storage/mill building and attached southern building addition on the Harrington's property (Appendix A, Figure 1). Please view Appendix A, Figure 2 for an overall demarcated Facility area to be included in this work plan. Tetra Tech will prepare addenda to the Voluntary Cleanup Plan for this Facility that will address specific elements of the SoW not included in this investigation. These elements will include cleanup activities on property owned Empire Building Materials, Inc. (EBM), the paved utility corridor along Wallace Avenue, and in the alley south of Heeb's. Additional remedies may be proposed in the addenda to address remaining contamination at the Facility.

The existing contamination on the EBM property, as investigated and presented in previous reports, will be addressed appropriately as part of the SI report where known Facility contamination and remedial remedies are discussed. As stated above, contamination on the EBM property will be addressed through addenda to the VCP. Any required additional soil investigation will be conducted at that time. The areas of known ore contamination are presented in Appendix A, Figure 3. Figure 3 indicates areas of asbestos ore on the EBM property near the South Warehouse, and areas south of the Harrington Property in the RoW. Asbestos ore is also known to be present under areas of the Heeb's parking lot and the alley directly to the south of Heeb's, although the extent of this contamination is currently undefined.

2.0 SUPPLEMENTAL INVESTIGATION WORK PLAN

Tetra Tech has prepared the following six tasks to-be addressed in the SI: Task No. 1 (Prepare a Brief Site Characterization Summary); Task No. 2 (Develop a Quality Assurance Project Plan (QAPP)); Task No. 3 (Prepare a Site Specific Health and Safety Plan); Task No. 4 (Develop a strategy to evaluate the condition of pavement in the following areas: previously uninvestigated areas on property owned by Harrington's, Inc., Empire Building Materials, along the corner of Wallace Avenue and Main Street, and in the alley south of Heeb's where asbestos ore may be located, including any paved and unpaved areas; Task No. 5 (Develop A Comprehensive Air-Related Assessment to address the interior of the two structures located on the Harrington's Property); and Task No. 6 (Develop a SI Report). Pending further investigation, Tetra Tech understands that additional areas may need to be addressed as revisions to this SI. Please view Appendix A, Figure 2 for a visual identification of additional investigation areas. Figures 1 – 4 in Appendix A indicate areas demarcated "Previously Assessed Area." This line indicates areas where excavation of contaminated soils has taken place and also areas that have been previously investigated. This line is representative, based upon previous documentation, of the boundary of assessed and/or remediated areas.

2.1 Site Characterization Summary

The additional properties to be addressed in this SI are part of the existing DEQ Comprehensive Environmental Cleanup and Responsibility Act (CECRA) -listed Facility. Portions of these additional properties not owned by the City must first allow for owner consent for access to their property to allow for this additional investigation. The City will exercise its best efforts to obtain access to the properties in order to conduct the investigation. If owner consent is not achieved, the City will request assistance from DEQ to obtain access to each property.

As part of this SI work plan, the City will request access from the owners of the Harrington property, Heeb's property, Empire Building Materials, Inc. property, and Story Distributing, Inc. property to accurately characterize the site. On behalf of the City, Tetra Tech will ask permission from the property owners using the access agreement as provided in Appendix B.

A comprehensive site characterization summary of the CMC Bozeman Facility is located in Section 4.0: *Environmental Assessment in the Voluntary Cleanup Plan for the CMC East Main Depot* (RTI, 2002) (VCP), and is incorporated herein by reference. Previous investigation along the right-of-way west of EBM and west of Story Distributing Property by Resource Technologies, Inc. (RTI) revealed visible asbestos contamination present beneath the roadway to a depth approximately no greater than eight inches from the top of the curb into the street as documented in Revision 1, *Addendum to the Voluntary Cleanup Plan for the CMC East Main Depot Facility, Bozeman, Montana* (RTI, 2003), and as incorporated herein by reference. In Tetra Tech's *Limited Soil Investigation Results Report* (as incorporated herein by reference) for the Nash-Finch / Bozeman Public Library property, dated July 5, 2007, compacted sand and gravel backfill was encountered above the native dark brown silty clay material at depth's from 0 – 4 feet in the four bore holes. The intent of the July 2007 investigation was to evaluate the presence of lead in native soil under paved parking areas. Note that the presence of asbestos ore was not evaluated, as previous investigations have shown no detectable asbestos in this area of the Facility. During the week of August 27, 2007, asbestos ore was discovered by Montana Department of Transportation (MDT) along the right-of-way along the west corner of Main Street and Wallace Avenue (adjacent to Heeb's). This asbestos ore was found during a recent MDT project to remove and replace existing sidewalk, curb, and gutter along Main Street; the asbestos ore appeared to be used as backfill material beneath these removed surfaces.

The asbestos ore appeared to extend beneath the Heeb's north-facing parking lot, under the sidewalk to the west along Main Street, and remains under the newly paved corner of Wallace Avenue and Main Street. During utility work conducted on behalf of the City of Bozeman on October 9, 2007, additional asbestos ore was found in the unpaved alley behind, and the south of, Heeb's. The extent of contamination in the unpaved alley behind Heeb's will be investigated as part of this SI and these additional asbestos ore discoveries will be addressed in an addendum to the VCP.

2.2 Supplemental Investigation Work Plan

Two separate characterizations will commence at the site and will address specifically the potential for asbestos in Facility soils and air-related asbestos inside the structures located on the Harrington property. The ambient air, dust, soil, and pavement condition evaluation methodologies are present for reference in Table I.

TABLE I: SAMPLE COLLECTION, PREPARATION, AND ANALYSIS
CMC Bozeman Facility Supplemental Investigation

Medium	Collection/Preparation	Analytical Method
Air	ISO Method 10312	TEM
Dust	ASTM Method D5755-03	TEM
Soil	CARB Method 435	PLM with QC @ 10% TEM
Pavement	Visual Pavement Condition Index	ASTM D5340-04

Historical documentation indicates that the original Harrington building may have been utilized for asbestos storage and/or milling during asbestos operations at the Facility. The possibility exists that any asbestos fibers present in the original building may have migrated or aerosolized (suspension of particles in air) into the more recently constructed building.

2.2.1 Asbestos Soil Sampling

Asbestos soil samples will be collected for this SI to document levels of anthophyllite in soils throughout the site in locations that have not been previously addressed. Areas where soil samples will be collected are discussed in Section 2.2.1.1. Specific areas where soils containing asbestos ore are known or likely to be present will be addressed through addenda to the VCP. The first addendum to the VCP will provide for excavation and encapsulation in the Wallace Avenue utility corridor, removal of asbestos contamination under Wallace Avenue, removal of any asbestos contamination found through this SI in areas adjacent to the Harrington's southern building addition (as depicted in Appendix A, Figure 4), removal of accessible asbestos contamination on Story Distributing, Inc. property, Empire Building Materials, Inc. property, the alley south of Heeb's, and any required confirmation sampling. A second addendum to the VCP will address any remaining areas of contamination at the Facility.

2.2.1.1 Objectives

Tetra Tech proposes to collect soil samples from the areas delineated in Appendix A, Figure 2. These areas include the alley to the south of Heeb's; paved areas to the west, 15' to the south, and along the east side of the south Harrington building. Contamination on the EBM property will be addressed in an addendum to the VCP. Tetra Tech will perform an evaluation of the existing pavement conditions, given current property uses, and potential for future required utility work. The evaluation will include use of a modified version of American Standard for Testing and Materials (ASTM) Method D5340-04 *Standard Test Method for Airport Pavement Condition Index Surveys*. This test method covers the determination of airport pavement condition through visual surveys of asphalt-surfaced pavements, including porous friction courses, and plain or reinforced jointed Portland cement concrete pavements, using the Pavement Condition Index (PCI) method of quantifying pavement condition. ASTM Method D5340-04 is found in Appendix C. Tetra Tech will use the PCI to help determine the relative condition of the pavements (i.e. for pavements that are deemed to be damaged the risk of sub-grade asbestos ore exposure is greater).

Pavement areas to be addressed are located in Appendix A, Figure 5, and primarily includes Wallace Avenue (from Main Street to Curtiss Street) including but not limited to areas beneath sidewalks and curbs, alleys that may contain pavement, and the parking lots of Heeb's and Harrington's.

Due to the possibility that asbestos ore materials may be encountered in utility corridors and during the excavation/utility repair work in Wallace Avenue, Tetra Tech will address these future work efforts in an addendum to the VCP to protect the health and safety of the utility workers and the community from potential asbestos exposure. Appendix D contains all utility maps that have been provided including City of Bozeman water main locations, City of Bozeman sewer locations, Northwestern Energy gas utility locations (electricity is from overhead power lines), and Qwest.

Generally, exterior asbestos soil sampling will consist of completing a visual assessment and/or using test pit soil sampling methodologies. The visual assessment will be completed for all test pit locations and will consist of documenting visible asbestos ore throughout the stratification layers of soil. If asbestos ore is noted through visual inspection, Tetra Tech will not collect a sample and will document to-depth findings. If visible asbestos ore is not noted, asbestos soil samples will be collected from each vertical and horizontal extent of each excavation from defined depth intervals. In this instance the vertical extent is defined as the bottom of the test pit from which one grab sample will be collected. The horizontal extent is identified as the northerly, southerly, easterly, and westerly locations along the test pit walls from which a sample will be composited. The composite sample will consist of soil collected at 6" intervals to the bottom of the completed test pit. Prior to test pit excavation, Tetra Tech will request utility locate to be performed throughout the SI area to prevent safety hazards.

Asbestos ore has been found in Facility soils at the surface and from depths of approximately 2 inches to 3 feet beneath ground surface (bgs). The asbestos ore is often mixed with native soil and/or fill material. Tetra Tech will subcontract a licensed and insured excavation company to use a backhoe for the collection of soil samples from a minimum depth of 3 feet into native soil. If visible asbestos is present at 3 feet below native soil contact, excavation will continue until visible asbestos is no longer present. Please view Appendix A, Figure 4 for proposed test pit locations in this SI work plan. The test pits are proposed to be located next to the Harrington's building intermittently spread 20' apart to the boundary of previously investigated or excavated areas and the east side of the south Harrington's building itself. As shown, Tetra Tech proposes to collect 13 composite and 13 grab samples from the test pits at this location. Approximately five test pits will be excavated in the alley south of Heeb's, with collection of approximately 5 composite and 5 grab samples.

After test pit sample collection, Tetra Tech's subcontracted excavation company will backfill and compact each testpit with the native exhumed material.

2.2.1.2 Sample Collection/Chain of Custody

Asbestos soil samples will be collected from excavation walls at every six inches of vertical depth for each test pit in pre-labeled plastic sampling containers. The samples will be viewed and a determination will be made if visible asbestos ore is present per each 6". Duplicate samples will be collected during sample collection and a composite sample of the entire vertical depth will be made in a quart-sized plastic container with the same amount of soil collected from each 6" subsample. The duplicates will be collected and stored in quart-sized plastic containers for each 6" subsample. Duplicate samples will be prepared and collected along with the original lab samples by using clean mixing bowls and hand trowels while "stirring" the collected soil for each sample collected. In-between each test pit location, Tetra Tech will don a new pair of nitrile gloves and will clean the mixing bowl and hand trowel to prevent cross-contamination.

Tetra Tech will use ASTM D-2487-92 and ASTM D-2488-93 to document each test pit on a standard test pit log form. Additionally, Tetra Tech will document each test pit location using a Global Positioning System (GPS), appropriate measurements in reference to existing structures, and pictorial documentation with each sample identification number. Appendix E contains Tetra Tech's standard test pit log.

After the collection of samples from each test pit, Tetra Tech will rinse and clean off the backhoe bucket using water into the original test pit.

Following the collection of samples, chain-of-custody procedures will be followed to establish a written record of sample handling and movement between the sampling site and the laboratory. Each shipping container will have a chain-of-custody form completed in duplicate by sampling personnel. Tetra Tech, Inc. will keep one copy of this form and the other copy will be sent to the laboratory. The chain-of-custody will contain the following information:

- Sample identification number;
- Sample collector's printed name and signature;
- Date and time of collection;
- Place and address of collection;
- Sample matrix;
- Analyses requested;
- Signatures of individuals involved in the chain of possession; and
- Inclusive dates of possession.

The chain-of-custody documentation will be placed inside the shipping container so that it will be immediately apparent to the laboratory personnel receiving the container, but will not be damaged or lost during transport. The shipping container will be sealed so that it will be obvious if the seal has been tampered with or broken.

2.2.1.3 Soil Sample Analysis

The asbestos content in soils will be determined through visual inspection and using a modified "bore hole sampling methodology" of California Air Resources Board (CARB) Method 435: Determination of Asbestos content of Serpentine Aggregate (Appendix F). This preparation methodology states that a minimum of three samples must be submitted per "area" sampled.

This soil analysis method was chosen to document the asbestos content in soils. Per the method, the composited sample:

> …shall be crushed to produce a material with a nominal size of less than three-eighths of an inch. Before crushing, the sample

> must be adequately dried. ASTM Method C-702-80, which is incorporated herein by reference, shall be used to reduce the size of the crushed grab sample to a one pint aliquot. The one pint aliquot shall be further crushed using a Braun mill or equivalent to produce a material of which the majority shall be less than 200 Tyler mesh…

Soil samples will be analyzed using the CARB Method 435 preparation, followed by analysis using polarized light microscopy (PLM) to 0.25%. The CARB Method 435 contains an abbreviated PLM analysis, but the more comprehensive PLM analysis outlined in EPA Method 600/R-93/116 will be utilized; Appendix G contains this Method.

Ten percent of soil samples collected will be analyzed as specified above, but also analyzed using transmission electron microscopy (TEM), with the CARB 435 preparation method, to 0.10%.

2.2.1.4 Quality Control

For the 18 composite samples to be collected (if necessary) from the side walls of the test pits, one duplicate sample for the complete side wall set will be submitted to the laboratory for analysis; additionally, one blank sample will be submitted to the laboratory for the complete side wall assessment. The blank sample will consist of using soil that has been previously analytically tested for asbestos and is known not to contain asbestos. The purpose of this sample collection is to ensure the laboratory does not enter sample contaminants into their sample handling procedures as well as to show that Tetra Tech does not enter contamination by route of sample handling into its sample collection process. These blank samples will increase the sample collection validity and accuracy for this SI.

For the 18 grab samples to be collected from the bottom of the test pits, one duplicate sample for the complete set of bottom samples will be submitted to the laboratory for analysis; additionally, one blank sample will be submitted to the laboratory.

2.2.2 Asbestos Air-Related Sampling

It is believed that the original Harrington's building, located in the 2000 block of S. Wallace, Avenue, was used as an asbestos ore storage building and/or for the processing of asbestos ore. Objectives of sampling are to define the nature and extent of any anthophyllite asbestos contamination of the indoor air in the Harrington's structures, and to determine the exposure potential to those who utilize the building.

2.2.2.1 Objectives

To determine if air-related surficial anthophyllite asbestos contamination is located in Harrington's buildings located in the SI area, Tetra Tech will collect dust samples using ASTM D 5755-03 and air samples in accordance with International Organization for Standardization (ISO) Method 10312:1995. Tetra Tech will collect dust samples in specified zones in both buildings using ASTM D 5755-03. Surficial deposition of any potential anthophyllite-contaminated dust will have settled in areas on the buildings with which no air movement is provided (corners, behind doors, tops of cabinets/shelves, etc…). Based on recent information indicating a lack of correlation between asbestos concentrations in dust samples and concentrations in air, the EPA has determined that the collection of dust samples alone will not adequately assess potential indoor sources of exposure. Due to this determination, Tetra Tech will also collect activity-based air samples during building owner/employer work activities throughout the workday to determine if and at what levels tenants, employees, or patrons to these building may be exposed to anthophyllite asbestos from residual contamination.

2.2.2.2 Asbestos Dust Sampling

For the use of ASTM D 5755-03, *Standard Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Structure Number Surface Loading* (Appendix H), Tetra Tech will collect a minimum of one composite sample per building with the following parameters:

- One composite sample will consist of 10 subsamples (aliquots) per work area in the building (a work area is defined as a segregated section in the building (e.g., office space, shop, etc.)) using the following ratio of subsample locations:
  - (4) accessible areas,
  - (4) infrequently accessed areas, and,
  - (2) inaccessible areas;

  Note: the sample order will include collection of one subsample initially from each of these areas (i.e. (1) accessible location, (1) infrequently access area, and (1) inaccessible area so if filter loading occurs the collected sample will be representative of all three areas.
- One composite sample will be collected for each floor of each building on the property;
- One composite sample will be collected for each separate heating, ventilation, and air conditioning (HVAC) system per floor of each zone in each building per property; and,
- One composite sample will be collected inside the return plenum of each HVAC zone in each building. If no HVAC is present, samples will be collected near negative air-induced entry points into the building in laminar/stagnant air locations (e.g., beneath window sills, in corners of building areas, beneath floor mats, etc.).

A subsample will consist of collecting ten (10) 100 centimeter (cm) x 100 cm samples throughout these defined composited spaces. A single template will be used for each composite sample; therefore, per each aliquot in the composite sample, a single template will be used.

Tetra Tech's decontamination procedure with consist of disposal of plastic gloves per composite sampling event, disposal of the template per composite sampling event, and using separate cassettes in-between each composite sampling event.

Although this analytical method's sensitivity is generally around 1,000 structures per square centimeter for use with a single (10cm x 10cm) template, Tetra Tech would like to increase the target analytical sensitivity to 20 structures per square centimeter based upon increased aliquot points – as discussed above - in the initial sampling. While ASTM D 5755-95 requires the identification of fibers with a 5:1 aspect ratio, Tetra Tech will request an aspect ratio of 3:1 be analyzed by the laboratory due to recent research involving amphibole asbestos risks associated with fiber sizes less than 5 microns in length.

Samples will generally be collected from "dusty" areas that may not have been cleaned previously and may be collected from any horizontal surface in each composite area; however, "non-dusty" surfaces will be encountered and sampled (routinely cleaned areas along the floor, window sills, frequently vacuumed area, etc.). The type of each surface sampled will be documented as porous, semi-porous, or non-porous (carpet, wood, floor tile/waxed or varnished wood, respectively) and will be labeled on a floor plan figure and documented in the following manner:

- South Harrington Building, Composite Sample 10, Subsample 7, Porous Surface
    - SHB-10-7-P.

Tetra Tech field sheets will be used that specifically document each area in the building the sampling commenced, the level of the building, and the specific type of surface from which each sample is collected.

More specifically, Tetra Tech will collect samples from the following composite locations with subsample identification to occur onsite:

- North Harrington Building
    - Basement
        - Shop/Office/Vault
        - HVAC Room
        - Showroom
    - Main Level
        - Bitterroot Stained Glass Shop and Storage Room
        - Main Office/Storage Room No. 1 & 2
        - Showroom/Closet
        - East Dock Area
        - Southwest Office/Men's and Women's Restroom
    - Attic Level
        - South Showroom
        - North Showroom
        - Breakroom/East Office/Hallway
    - 3rd Level
        - Big Sky Aikido
- South Harrington Building
    - Salvation Army Shop & Bathroom

Appendix A, Figures 6 – 10 represent these sample locations.

Analysis will indicate the type of asbestos (if any) present on each composite area analyzed. Analysis will be completed using transmission electron microscopy (TEM). This analysis will differentiate between specific types of asbestos materials, thus allowing the City to effectively determine if contamination from asbestos ore mining operations has contaminated the surface of indoor areas.

2.2.2.2 Asbestos Air Sampling

For the use of ISO Method 10312:1995, *Ambient Air – Determination of Asbestos Fibres – direct Transmission Electron Microscopy Method* (Appendix I), Tetra Tech will collect activity-based air samples throughout two eight hour work periods via personal air monitoring of two employees at the Harrington's North building during an average work day, or through activity-based sampling conducted by Tetra Tech, Inc. simulating daily activities of Harrington's, Inc. employees. During a September 12, 2007, site visit, Jeff Harrington indicated he would prefer that he and another employee participate in sample collection. Typical daily work activities (tasks), according to Mr. Harrington, will include the following tasks: cleaning furniture, moving furniture, furniture sales, office work, limited janitorial duties, and management duties (including observing employees performing all above-mentioned duties). Should Mr. Harrington and/or his employee decline to participate at the time of sample collection, a Tetra Tech, Inc. employee will conduct activity-based sampling inclusive of the activities listed at the direction of Mr. Harrington.

The 25 millimeter (mm) conductive cowl cassette with 0.45 micron pore size mixed cellulose ester (MCE) sampling filter backed by a 5.0 micron pore size MCE filter will be worn by the employees within seven inches of the mouth/nose area (such as on the lapel of their clothing) throughout the duration of the sampling period. Prior to and after sample collection the flow rate of the sampling pump will be calibrated to 2.0 liters per minute (lpm) using a primary source. If samples are found to be out of tolerance by +/- 10% of the initial calibrated flow rate, the sample will be considered void and a retest will be performed.

The analysis will be conducted using transmission electron microscopy (TEM) by a laboratory participating at a minimum in the National Voluntary Laboratory Accreditation Program (NVLAP) and conducted in accordance with the specifications found in ISO 10312 including but not limited to the following procedures:

- Analytical Sensitivity (0.0002 cc^{-1});
- Filter Overload Target set a 10% with the laboratory notifying Tetra Tech, Inc. of any overloaded filters prior to proceeding with analysis;
- Counting Rules specifying that the laboratory read a minimum of 10 grid openings and continue to count structures until the required analytical sensitivity has been reached, based on the sample volume and the number of grid openings counted. The count may be terminated upon completion of the grid opening containing the 50^{th} structure, regardless of whether or not the target analytical sensitivity has been reached; and,
- A media blank and a field blank from the same sample media lot will be submitted to the laboratory for quality control.

All field equipment will be decontaminated using wet wipes prior to the start of sampling, in-between sampling events, and after sampling events to prevent contamination of the equipment.

2.2.2.3 Sample Handling/Chain of Custody

Samples will be handled in accordance with ASTM D5755-03, which will include donning a new pair of nitrile gloves between composite sample locations prior to each sample. A chain-of-custody will be filled out for each composite sample and will indicate all aliquot sample identification numbers included for that sample.

Documentation of each sample area will consist of location of each sample aliquot on a building floor plan as well as a pictorial representation of sample location with each individual aliquot number.

2.2.2.4 Dust and Air Sample Analysis

The dust samples will be collected and submitted for analysis for identification of asbestos fibers in accordance with ASTM D 5755-03 and ISO Method 10312:1995 for the air samples. Analysis will be completed by EMSL Analytical Laboratories in West Mont, New Jersey, for all samples. EMSL is accredited by the American Industrial Hygiene Association (AIHA) for asbestos analysis and participates in NVLAP. By use of a TEM for all dust and air samples analyzed, Tetra Tech will be able to differentiate between the different types of asbestos present on the sample medium, an important trait due to the specific morphology and crystallization of the Karst Mine anthophyllite from differing asbestos structures.

2.2.2.5 Quality Control

One media blank sample will be submitted to the laboratory for analysis for at least 10% of the indoor air samples collected in accordance with ASTM D 5755-03. Additionally, one field blank sample will be submitted to the laboratory for 10% of the samples collected. The field blank sample will be collected by allowing the same sample volume 2.0 liters per minute of flow for a period of 30 seconds in the air for the dust samples.

One media blank sample and one field blank sample will be submitted for analysis for ISO Method 10312 to meet a minimum suggested level of 20% for the laboratory samples submitted. Additionally, prior to the sampling, ISO Method 10312 suggests that two cassettes from the lot of provided samples be submitted to a laboratory for TEM analysis to determine the mean asbestos structure count. If the mean count for all types of asbestos structures is found to be more than 10 structures/square millimeter, or if the mean fiber count for asbestos fibers and bundles longer than 5 microns is more than 0.1 fibers per square millimeter, then the media lot will be rejected.

2.2.3 Laboratory Analytical Protocol

Tetra Tech will use a laboratory that participates with the National Voluntary Laboratory Accreditation Program (NVLAP) to analyze all samples in accordance with respective analytical laboratory methodologies.

2.3 Quality Assurance Project Plan

Procedures described in this section are designed to guide quality assurance. This section presents a discussion of the SI Work Plan goals to ensure data validity throughout the sample collection and analysis specifically for this Work Plan.

2.3.1 Sampling Design

Section 2.2 details sampling protocols, including the types and numbers of samples, based on review of historic data and previous investigations completed at the Facility. The sampling design for the various media is described below.

2.3.1.1 Pavement Condition Evaluation

The visual assessment of pavement currently found in the SI Work Plan focus area in the Facility is used to characterize the degradation of current pavement conditions.

2.3.1.2 Asbestos Soil Sampling

Test pit asbestos soil sampling will be used to estimate the quantity of anthophyllite in soil present in the study area.

2.3.1.3 Asbestos Air Sampling (Interior Dust Samples)

Interior dust samples in the Harrington’s buildings will define if the presence of anthophyllite structures exists along horizontal surfaces inside each of the buildings. Note that the North Harrington building has been documented to store asbestos ore and the South Harrington building has been documented as possibly having asbestos ore backfill material beneath the structure.

2.3.1.4 Asbestos Air Sampling (Interior Worker Task Sampling)

Interior worker task sampling will identify if throughout the course of a work day, Harrington's Furniture employees are exposed to anthophyllite asbestos present in the air. This sampling will identify if, during the course of the work day, air velocity surrounding horizontal surfaces or use of the Heating, Ventilation, and Air Conditioning (HVAC) system may aerosolize and suspend particulate matter in the air.

2.3.2 Measurement Data Acquisition

The type and quantity of samples, sampling methods, sample handling, chain-of-custody procedures, and analytical methods required for field investigations at the Facility are described in Section 2.2.

2.3.2.1 Field Quality Control Sampling

For the asbestos soil sampling, one soil duplicate will be submitted to the laboratory for analysis from one of the scheduled test pits. Additionally, one soil blank sample, a previously submitted soil sample known to contain no asbestos, will be submitted to the same laboratory for analysis. Of the samples collected from the bottom of the test pits, a single duplicate grab sample and a blank sample will be collected and submitted for laboratory analysis.

Prior to the collection of the asbestos dust samples, two media blank samples from the lot of filter media cassettes will be analyzed in accordance with sample methodology to ensure that these specific media are not contaminated with asbestos fibers. During the collection of the dust samples, and in accordance with the method, Tetra Tech will submit one blank for 10% of the dust samples collected. This sample will be collected by uncapping the filter cassette cap and allowing it to be exposed to building air for 30 seconds, thus acting as a field blank and providing sampler error information.

Prior to the collection of the asbestos air samples, two media blank samples from the lot of filter media cassettes will be analyzed in accordance with sample methodology to ensure that these specific media are not contaminated with asbestos fibers. During the collection of the dust samples, Tetra Tech will submit one blank for 20% of the dust samples collected. This sample will be collected by collecting 30 seconds worth of volume from the sample directly from the air.

2.3.2.2 Laboratory Quality Control

The laboratory chosen to analyze all asbestos samples will participate in the NVLAP. Participation in this national program includes quarterly round-robin samples for analytical asbestos detection. These prepared samples include a known concentration of asbestos; when the laboratory performs analysis of these "spiked" samples their analysts must fall within the allowable standard deviation for the specific sample analyzed. Participation in this program ensures both precision and accuracy of both the equipment used in the analysis and the analysts themselves. By subcontracting a laboratory participating in NVLAP, Tetra Tech ensures analytical results are both precise and accurate.

2.3.2.3 Equipment Operation and Calibration

All field and laboratory equipment will be operated, maintained, and calibrated in accordance with applicable methodologies while using the manufacturer's recommended procedures. Section 2.2 details the analytical methods, which in turn specify the laboratory equipment operation, maintenance, and calibration procedures.

2.3.2.4 Data Management

The Tetra Tech project manager will be responsible for ensuring that project personnel have the most current version of this SI and other project planning documents. The Tetra Tech project manager will maintain project files and project documents in Tetra Tech's Great Falls, Montana, office.

Analytical data will be provided to Tetra Tech in both electronic and hard copies. Analytical laboratory data as well as pertinent field notes/data will be directly downloaded onto the Great Falls Server. During report generation and throughout the project, the Great Falls server is backed up daily to prevent loss of data during the data reduction process.

The letter report for this assessment will include field notes, field logs, field forms, chain-of-custody records, evaluation of data quality, and analytical reports. Tetra Tech will also report information that relates to decisions for subsequent assessment work and redevelopment.

Corrective actions will be taken immediately upon identification of potential problems with data acquisition or measurements. Field equipment malfunctions will be identified immediately and corrected by the field team leader. Corrective actions will be documented in the field notes.

Tetra Tech's project manager will perform internal quality assurance audits to ensure data collection and data management, including data review, verification, and validation are performed in accordance with the SI objectives. Validation of the collected data will be included in the report to DEQ.

2.4 Site Specific Health and Safety Plan

Tetra Tech will prepare a Site Specific Health and Safety Plan (HASP) for the SI based upon final comments from DEQ for the work plan due to any revisions DEQ may suggest with the work plan. While DEQ does not provide approval of the HASP, Tetra Tech will supply DEQ with the finalized HASP for this SI prior to the start of any onsite work. The HASP will conform to the requirement 29 CFR 1910.120, and will generally follow using the same health and safety principles as are documented in the *Health and Safety Plan for the CMC East Main Depot Site Voluntary Cleanup Bozeman, Montana*, (RTI, 2002) and as included in the *Voluntary Cleanup Plan for the CMC East Main Depot, Revision 2*, (RTI, 2002) and are included by reference herein. Upon DEQ's acceptance of the SI, Tetra Tech will complete the HASP accordingly (dates, subcontractors, etc.). Tetra Tech health & safety Standard Operating Procedures (HSSOP's) that will be referred in the HASP are will include:

- HSSOP-1 (Site Specific Health and Safety Plans);
- HSSOP-4 (Exposure Monitoring);
- HSSOP-7 (Hearing Protection);
- HSSOP-8 (Personal Protective Equipment);
- HSSOP-9 (Respiratory Protection);
- HSSOP-14 (Emergency Response and Fire Protection);
- HSSOP-16 (First Aid and CPR);
- HSSOP-19 (Safety Training for Supervisors);
- HSSOP-20 (Employee Drug and Alcohol Program);
- HSSOP-21 (Medical Records Access);
- HSSOP-24 (Ground Fault Interrupters and Electrical Safety);
- HSSOP-25 (Material Handling);
- HSSOP-27 (Heat and Cold Stress and Biological Agents);

- HSSOP-31 (Work Site Management);
- HSSOP-34 (Spill Cleanup);
- HSSOP-36 (Personnel/Equipment Decontamination Procedures); and,
- HSSOP-39 (Accident Investigation).

It should be noted that the HASP will require personnel and/or work zone-downwind ambient air monitoring during the exterior test pit sample collection and interior dust samples for the following contaminants (with respective methodology):

- Asbestos: National Institute of Occupational Safety and Health (NIOSH) Method 7400.

All personnel on site, including all subcontractors, will be trained as required by the Occupational Safety and Health Administration (OSHA) Hazardous Waste Operations and Emergency Response (HAZWOPER) 29 CFR 1910.120 standard and provide proof of such training. Due to the presence of potential hazardous substances on site, Tetra Tech will require excavation subcontractors to participate in a respiratory protection program as defined in 29 CFR 1910.134. As required under this OSHA standard, all personnel on site will be required to receive physician's approval before wearing negative pressure air-purifying respirators and receive fit testing for the specific respirators worn. Prior to the onset of work, Tetra Tech will inform and provide the drilling subcontractors with the HASP as well as necessary training prior to starting work**.**

2.5 SI Report Preparation

Tetra Tech will prepare a final report presenting the findings of the Supplemental Investigation. The report will include the following:

- A general introduction describing the purpose and organization of the report;
- A summary of the investigations conducted pursuant to the SI Work Plan including general field observation and any deviations from the SI Work Plan;
- All validated field and laboratory analytical results will be included in narrative text discussion and in an appendix;
- All field notes and photographs in an appendix;
- A presentation and evaluation of the results of the investigation or tests conducted pursuant to the SI Work Plan, as well as QA/QC results, in text discussion, tabular form, and illustrated on figures such as maps;
- An evaluation of the fate and transport of contamination from the known areas containing asbestos ore;
- A discussion of potential hazardous or deleterious substance migration routes and human receptors, including a comparison of contaminant concentrations to appropriate screening levels;
- A summary of any other pertinent information obtained during the Supplemental Investigation; and,
- Conclusions and recommendations for further remedial actions.

3.0 SCHEDULE

Upon approval from the DEQ, Tetra Tech and the City will coordinate implementation of the SI. Sampling will be conducted within 20 working days of receiving work plan approval, provided the ground is not muddy. Laboratory analytical results are typically available within four weeks of receipt of the samples at the laboratory. A draft letter report will be submitted to the City for review, and the approved draft submitted to DEQ within 45 days from receipt of analytical results for the SI sampling. The final SI report will be issued to DEQ within 20 days of the receipt of DEQ comments on the draft.

Revised SI Prepared By:
Tetra Tech

Keith Cron, CIH
Branch Manager

4.0 REFERENCES

Resource Technologies, Inc. (RTI) (2002) *Voluntary Cleanup Plan for the CMC East Main Depot Bozeman, Montana, Revision 2.* Prepared for the City of Bozeman. Report Dated October 2002.

Resource Technologies, Inc. (RTI) (2002) *Heath and Safety Plan for the CMC East Main Depot Site Voluntary Cleanup Bozeman, Montana.* Prepared for the City of Bozeman. Report Dated September 4, 2002.

Resource Technologies, Inc. (RTI) (2004) *Voluntary Cleanup and Redevelopment Act Voluntary Cleanup Completion Report, Revision 1, CMC East Main Depot Site The Primary Portion of the CMC Asbestos Bozeman Facility Bozeman, Montana.* Prepared for the City of Bozeman. Report Dated August 2004.

TETRA TECH

APPENDIX A:
Figures

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2005 Montana NAIP Aerial Photograph

NORTH

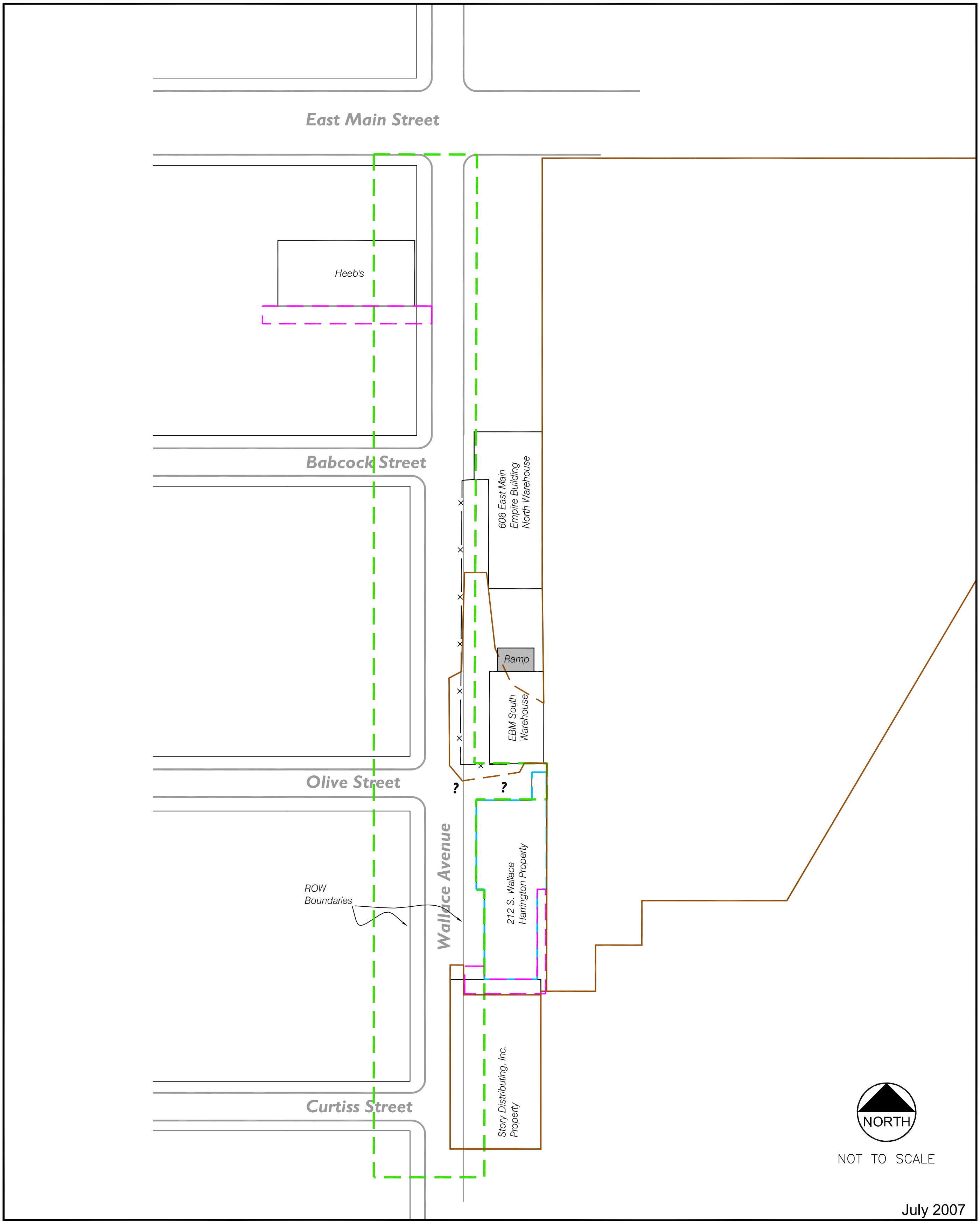
0 50 100 200
Feet

7720035.100

Approximate Previously Assessed Area

Approximate Area to be Included in SI

Appendix A
CMC Bozeman Facility
Bozeman, Montana
Figure 1



July 2007

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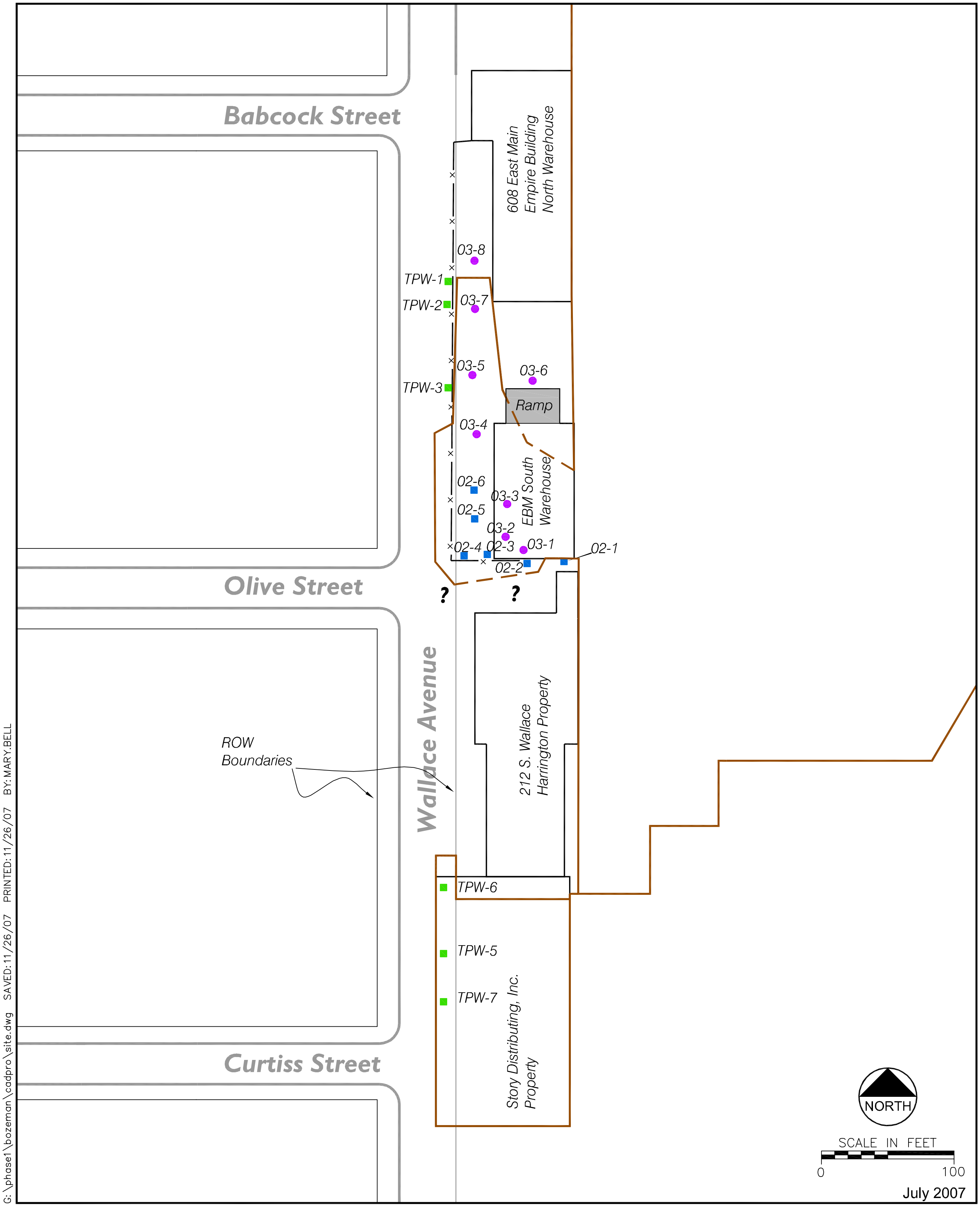
Interior Area to be included in SI
Exterior Area to be included in SI
Fence
Previously Assessed Area
Proposed Pavement Assessment Area

7720035.100

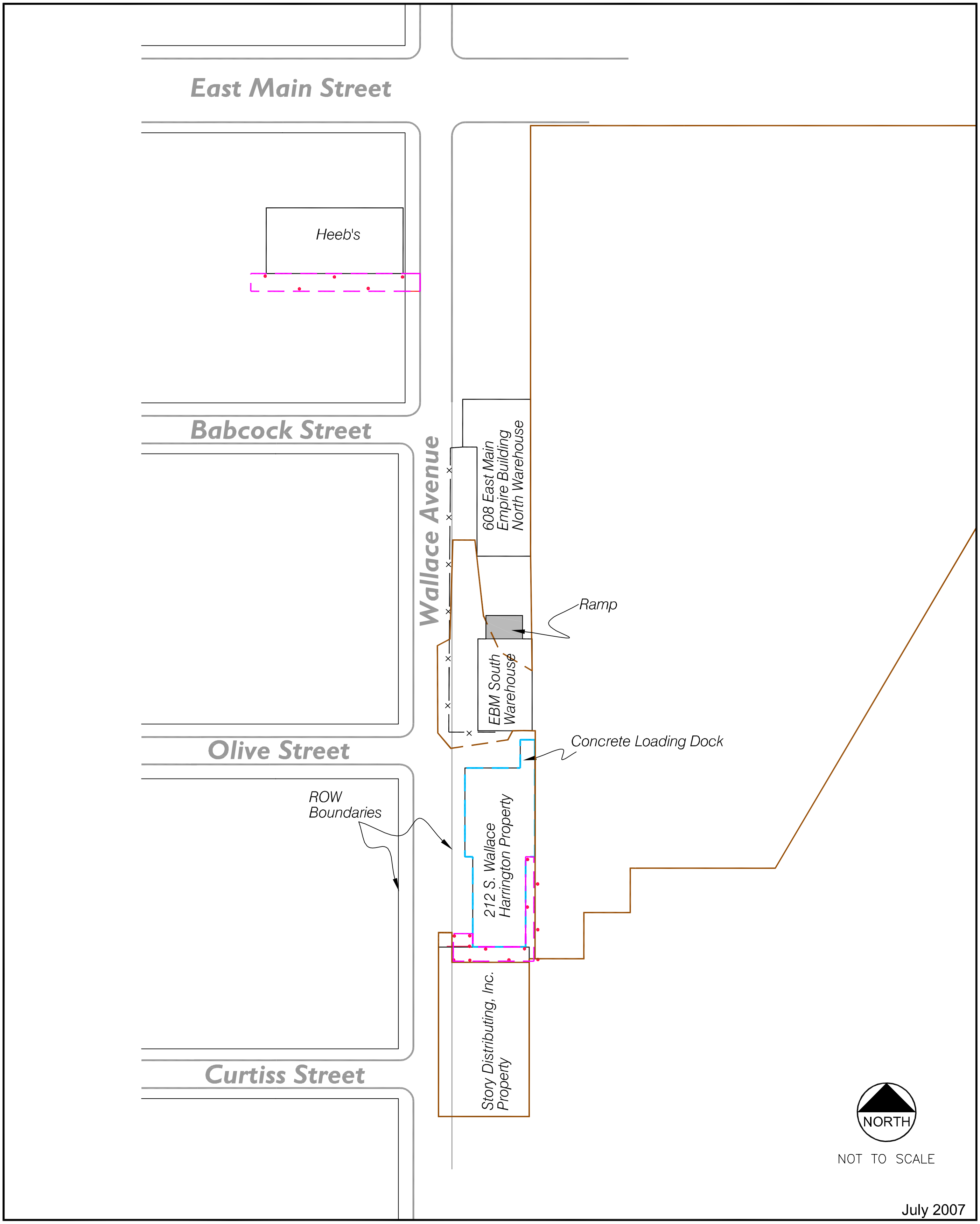
Appendix A
CMC Bozeman Facility
Bozeman, Montana
FIGURE 2



TPW-1 August 2003 Test Pit
02-1 October 2002 Test Pit Full Identification is TP02-#
03-1 April 2003 Test Pit Full Identification is EBM-03-#
Fence
Previously Assessed Area

7720035.100

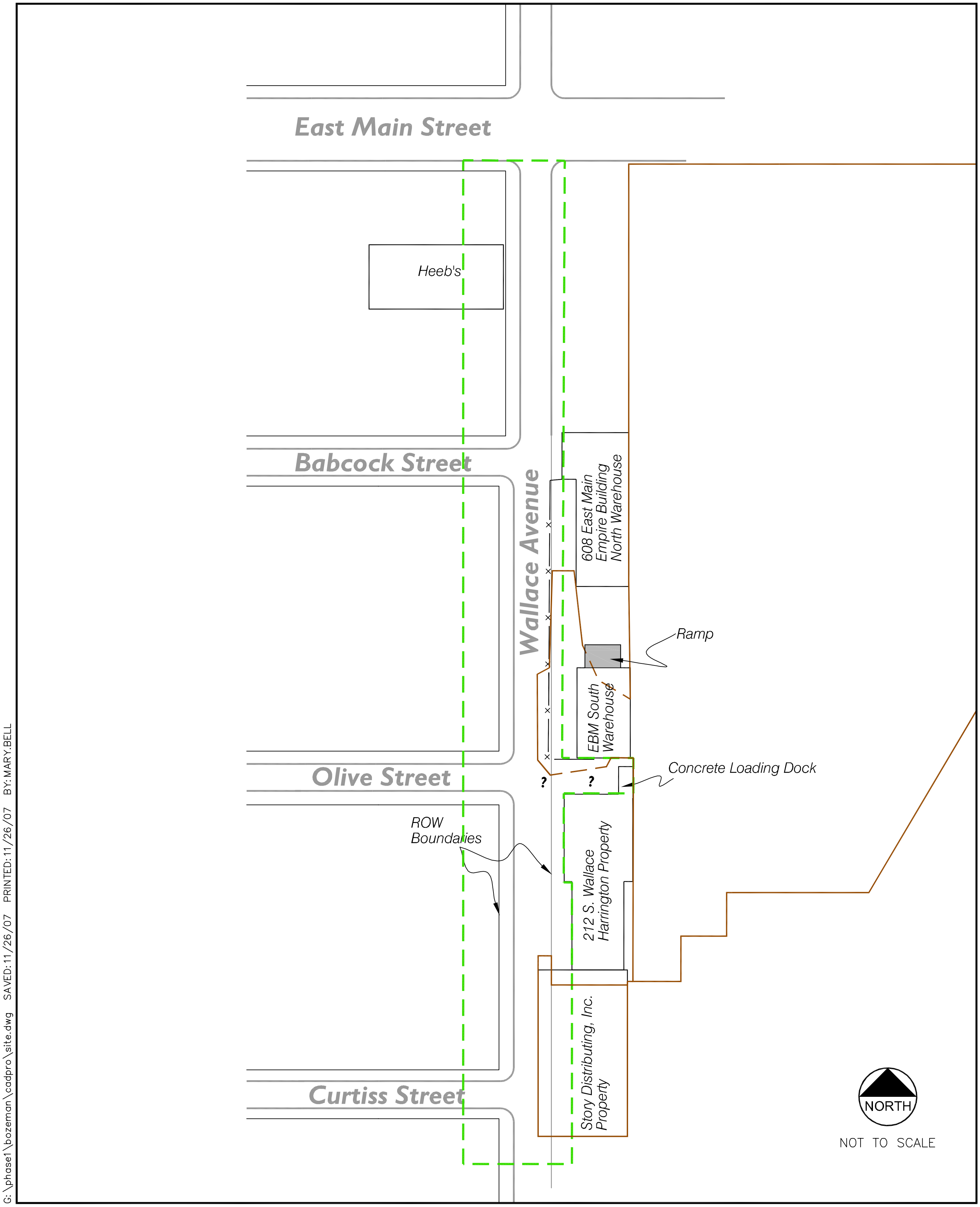
Appendix A
CMC Bozeman Facility
Bozeman, Montana
FIGURE 3



Interior Area to be included in SI
Exterior Area to be included in SI
Fence
Previously Assessed Area
Proposed Test Pit

7720035.100

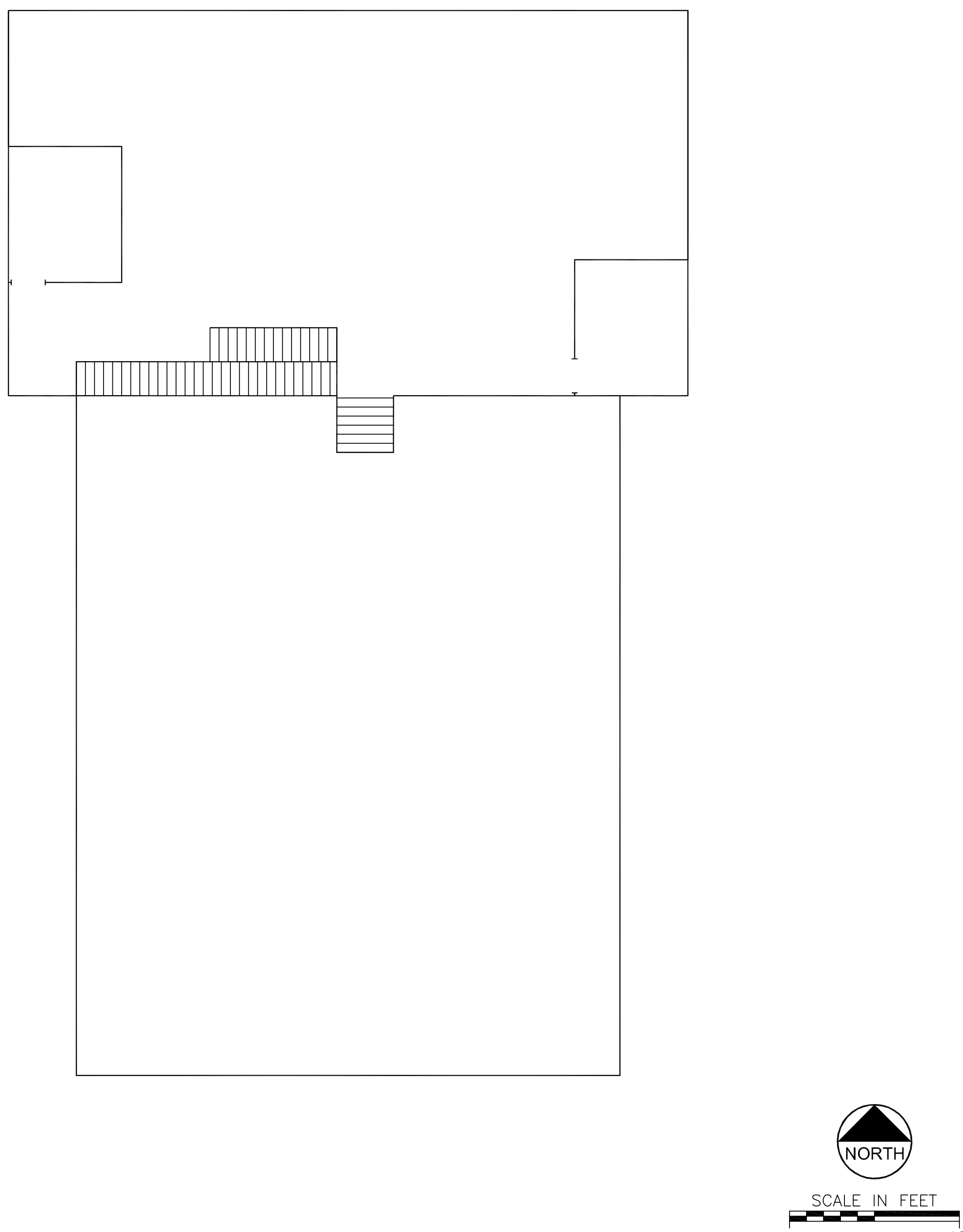
Appendix A
Proposed Test Pit Locations
CMC Bozeman Facility
Bozeman, Montana
FIGURE 4



Previously Assessed Area
Proposed Pavement Assessment Area

7720035.100

Appendix A
Pavement Assessment
CMC Bozeman Facility
Bozeman, Montana
FIGURE 5



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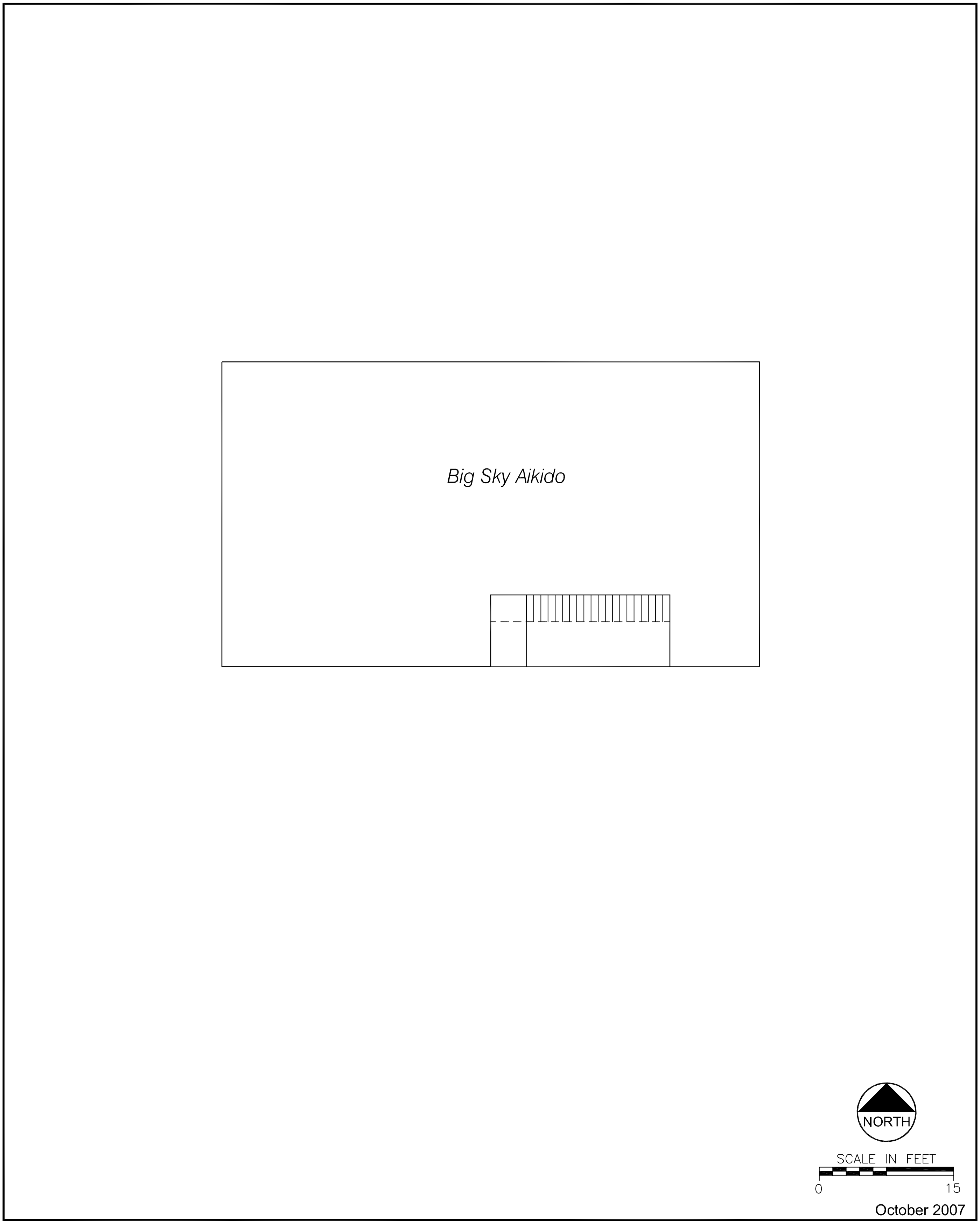
Attic Level - Harrington's
City of Bozeman - SI Workplan Development
Bozeman, Montana
FIGURE 6



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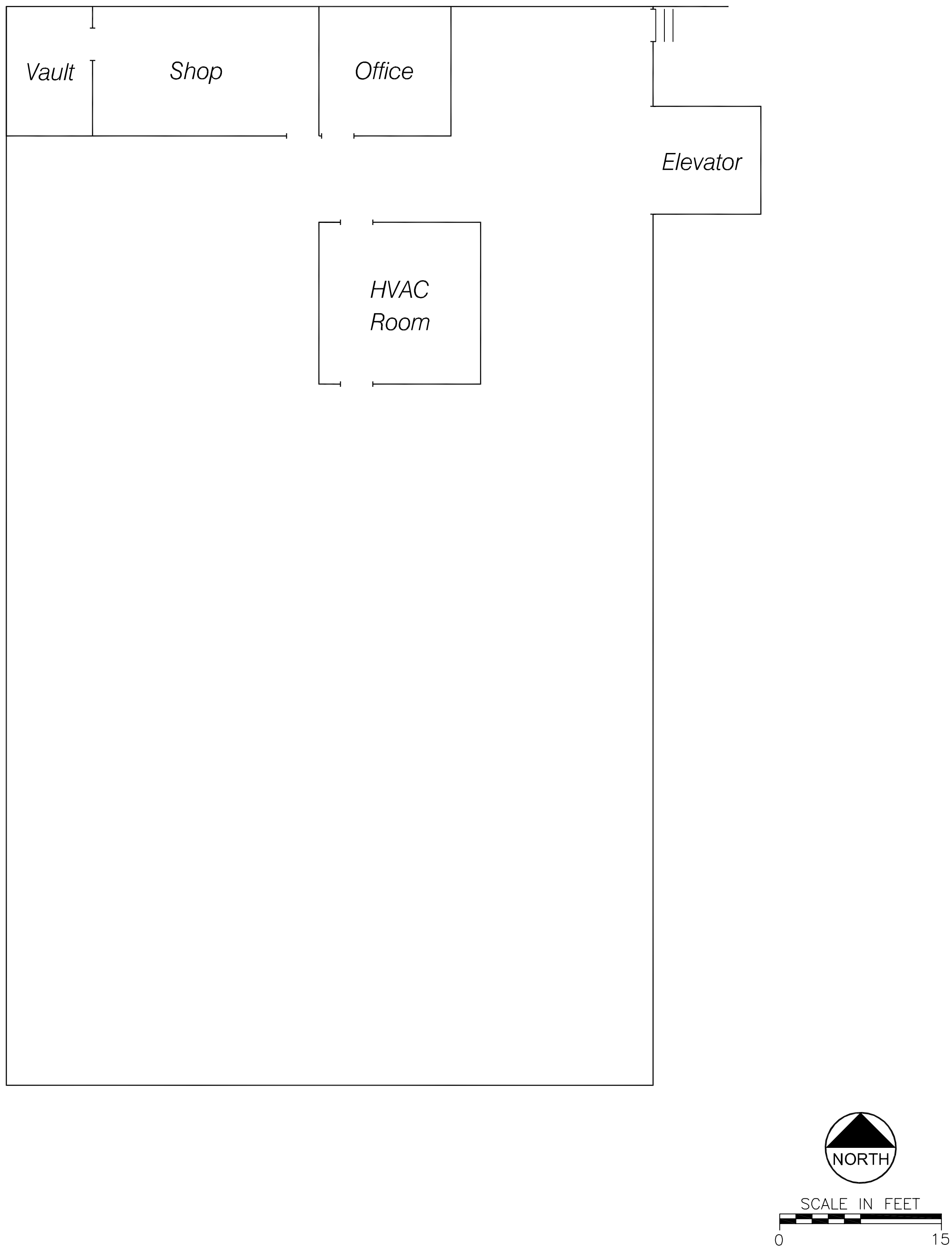
Third Level - Harrington's
City of Bozeman - SI Workplan Development
Bozeman, Montana
FIGURE 8



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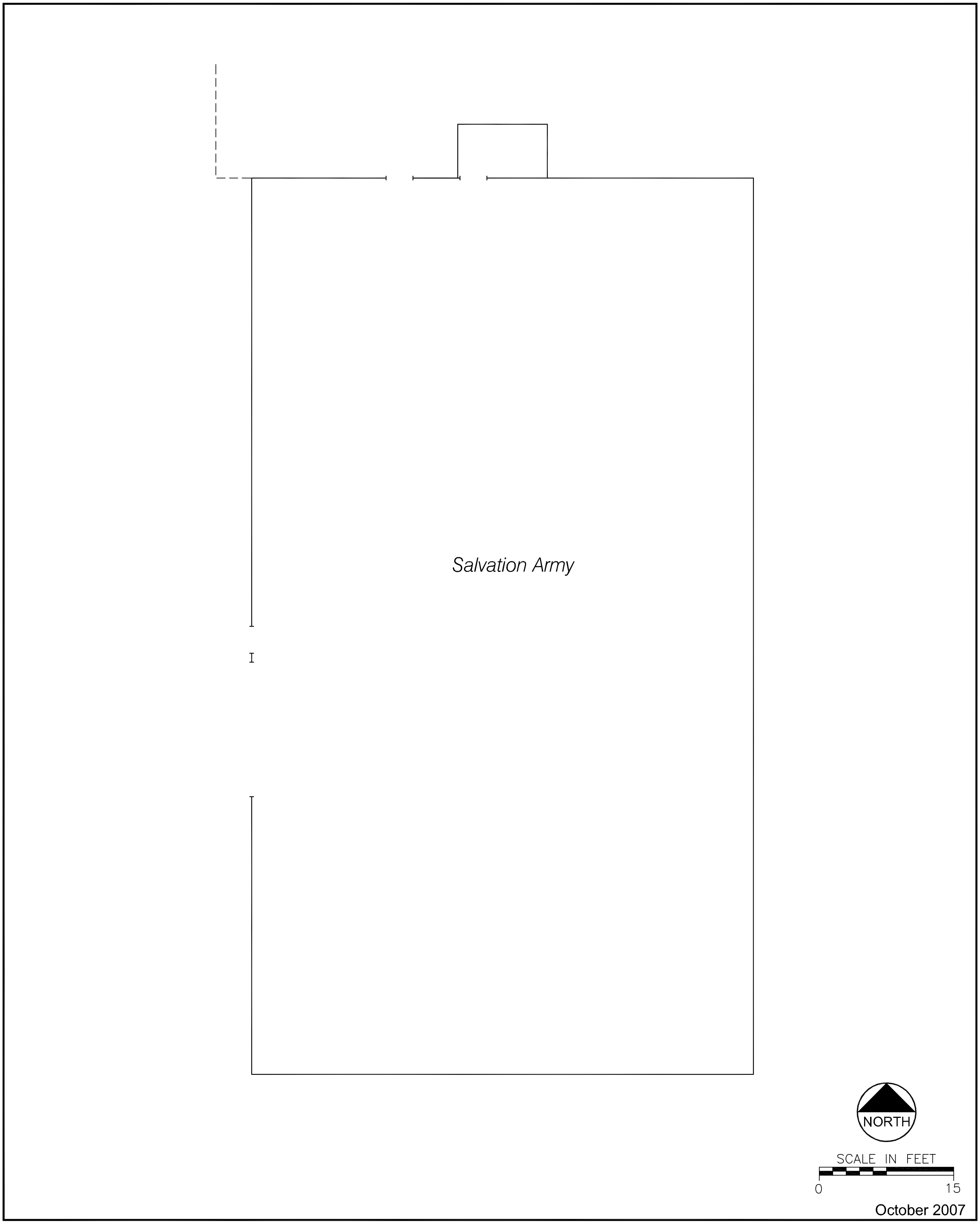
Basement of Harringtons
City of Bozeman - SI Workplan Development
Bozeman, Montana
FIGURE 9

7720035.100



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Salvation Army - South Harrington's Building
City of Bozeman - SI Workplan Development
Bozeman, Montana
FIGURE 10

APPENDIX B:
Written Consent of Property Owners for Supplemental Investigation Work Plan

WRITTEN CONSENT OF PROPERTY OWNERS FOR VOLUNTARY PROPERTY ACCESS TO:

{Name of Property}

Sections 75-10-730 through 738, Montana Code Annotated (MCA) requests the written consent of current property owners to be included as part of a voluntary cleanup plan. As part of the process for the cleanup efforts, the Montana Department of Environmental Quality (DEQ) has identified additional areas to be included in part with the voluntary cleanup plan efforts. To effectively determine if additional cleanup is needed, a Supplemental Investigation of asbestos-containing ore in and along your property must be completed.

Along the exterior of your property we request access to collect test pit samples along the west, south, and east portions of your property. Potentially, along the interior of your building, we request access to collect air and surficial dust samples.

Section 75-10-733(2)(c), MCA requires that voluntary cleanup plans must include, “The written consent of current owners of the facility or property to both the implementation of the voluntary cleanup plan and access to the facility by the applicant and its agents and the department.” The following agreement has been developed to satisfy this requirement.

As a property owner of {Name of Property} as described below, I, ____________, provide consent for the implementation of this voluntary cleanup plan proposed for the facility as approved by DEQ. I further grant access to the facility to Tetra Tech, its agents, and DEQ.

{Legal Property Description}

{Owner Name and Address}

____

Signature(s)

____

Name/Title (please print)

____

Date

TETRA TECH

APPENDIX C:
ASTM Method D5340-04

Designation: D 5340 – 04ε1

Standard Test Method for Airport Pavement Condition Index Surveys[1]

This standard is issued under the fixed designation D 5340; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

ε1 Note—Annex figures were corrected editorially in March 2005.

1. Scope

1.1 This test method covers the determination of airport pavement condition through visual surveys of asphalt-surfaced pavements, including porous friction courses, and plain or reinforced jointed portland cement concrete pavements, using the Pavement Condition Index (PCI) method of quantifying pavement condition.

1.2 The PCI for airport pavements was developed by the US Army Corps of Engineers through the funding provided by the U.S. Air Force (**1, 2, 3**).[2] It is further verified and adopted by FAA (**4**), and the U.S. Naval Facilities Engineering Command (**5**).

1.3 The values stated in inch-pound units are to be regarded as the standard. The SI units given in parentheses are for information only.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary statements are given in Section 6.

2. Terminology

2.1 *Definitions of Terms Specific to This Standard:*

2.1.1 *additional sample*—a sample unit inspected in addition to the random sample units to include nonrepresentative sample units in the determination of the pavement condition. This includes very poor or excellent samples that are not typical of the section and sample units which contain an unusual distress such as a utility cut. If a sample unit containing an unusual distress is chosen at random, it should be counted as an additional sample unit and another random sample unit should be chosen. If every sample unit is surveyed, then there are no additional sample units.

2.1.2 *asphalt concrete (AC) surface*—aggregate mixture with an asphalt cement binder. This term also refers to surfaces constructed of coal tars and natural tars for purposes of this test method.

2.1.3 *pavement branch*—a branch is an identifiable part of the pavement network that is a single entity and has a distinct function. For example, each runway, taxiway, and apron areas are separate branches.

2.1.4 *pavement condition index (PCI)*—a numerical rating of the pavement condition that ranges from 0 to 100 with 0 being the worst possible condition and 100 being the best possible condition.

2.1.5 *pavement condition rating*—a verbal description of pavement condition as a function of the PCI value that varies from "Failed" to "Excellent" as shown in Fig. 1.

2.1.6 *pavement distress*—external indicators of pavement deterioration caused by loading, environmental factors, or construction deficiencies, or a combination thereof. Typical distresses are cracks, rutting, and weathering of the pavement surface. Distress types and severity levels detailed in Appendix X1 for AC and Appendix X2 for PCC pavements must be used to obtain an accurate PCI value.

2.1.7 *pavement sample unit*—a subdivision of a pavement section that has a standard size range: 20 contiguous slabs (± 8 slabs if the total number of slabs in the section is not evenly divided by 20, or to accommodate specific field condition) for PCC airfield pavement and 5000 contiguous square feet ($\pm$ 2000 ft^2 (450 $\pm$ 180 m^2) if the pavement is not evenly divided by 5000, or to accommodate specific field condition) for AC airfield pavement and porous friction surfaces.

2.1.8 *pavement section*—a contiguous pavement area having uniform construction, maintenance, usage history, and condition. A section should also have the same traffic volume and load intensity.

2.1.9 *porous friction surfaces*—open-graded select aggregate mixture with an asphalt cement binder. This is a subset of asphalt concrete-surfaced pavements.

---

[1] This test method is under the jurisdiction of ASTM Committee E17 on Vehicle-Pavement Systems and is the direct responsibility of Subcommittee E17.41 on Pavement Management.

Current edition approved July 1, 2004. Published July 2004. Originally approved in 1998. Last previous edition approved in 2003 as D 5340 – 03.

[2] The boldface numbers in parentheses refer to a list of references at the end of the text.

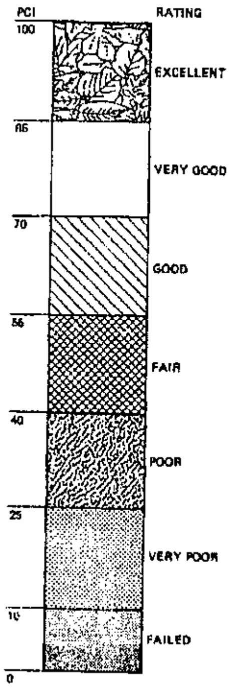


FIG. 1 Pavement Condition Index (PCI) and Rating Scale

2.1.10 *portland cement concrete (PCC) pavement*—aggregate mixture with portland cement binder including nonreinforced and reinforced jointed pavement.

2.1.11 *random sample*—a sample unit of the pavement section selected for inspection by random sampling techniques, such as a random number table or systematic random procedure.

3. Summary of Test Method

3.1 The pavement is divided into branches that are divided into sections. Each section is divided into sample units. The type and severity of airport pavement distress is assessed by visual inspection of the pavement sample units. The quantity of the distress is measured as described in Appendix X1 and Appendix X2. The distress data are used to calculate the PCI for each sample unit. The PCI of the pavement section is determined based on the PCI of the inspected sample units within the section.

4. Significance and Use

4.1 The PCI is a numerical indicator that rates the surface condition of the pavement. The PCI provides a measure of the present condition of the pavement based on the distress observed on the surface of the pavement which also indicates the structural integrity and surface operational condition (localized roughness and safety). The PCI cannot measure the structural capacity, neither does it provide direct measurement of skid resistance or roughness. It provides an objective and rational basis for determining maintenance and repair needs and priorities. Continuous monitoring of the PCI is used to establish the rate of pavement deterioration, which permits early identification of major rehabilitation needs. The PCI provides feedback on pavement performance for validation or improvement of current pavement design and maintenance procedures.

5. Apparatus

5.1 *Data Sheets*, or other field recording instruments that record at a minimum the following information: date, location, branch, section, sample unit size, slab number and size, distress types, severity levels, quantities, and names of surveyors. Example data sheets for AC and PCC pavements are shown in Fig. 2 and Fig. 3.

5.2 *Hand Odometer Wheel*, that reads to the nearest 0.1 ft (30 mm).

5.3 *Straightedge or String Line* (AC only), 10 ft (3 m).

5.4 *Scale*, 12 in. (300 mm) that reads to ⅛ in. (3 mm) or better. Additional 12-in. (300-mm) ruler or straightedge is needed to measure faulting in PCC pavements.

5.5 *Layout Plan*, for airport to be inspected.

6. Hazards

6.1 Traffic is a hazard as inspectors must walk on the pavement to perform the condition survey. Inspection must be approved by and coordinated with the airport operational staff.

6.2 Noise from aircraft can be a hazard. Hearing protection must be available to the inspector at all times when airside inspections are being performed.

7. Sampling and Sample Units

7.1 Identify areas of the pavement with different uses such as runways, taxiways, and aprons on the airport layout plan.

7.2 Divide each single-use area into sections based on the pavement design, construction history, traffic, and condition.

7.3 Divide the pavement sections into sample units. If the pavement slabs in PCC have joint spacings greater than 25 ft (8 m), subdivide each slab into imaginary slabs. The imaginary slabs should all be less than or equal to 25 ft (8 m) in length, and the imaginary joints dividing the slabs are assumed to be in perfect condition. This is needed because the deduct values were developed for jointed concrete slabs less than or equal to 25 ft (8 m).

7.4 Individual sample units to be inspected should be marked or identified in a manner to allow inspectors and quality control personnel to easily locate them on the pavement surface. Paint marks along the edge and sketches with locations connected to physical pavement features are acceptable. The use of nails or other potential FOD sources is not recommended. It is necessary to be able to accurately relocate the sample units to allow verification of current distress data, to examine changes in condition with time of a particular sample unit, and to enable future inspections of the same sample unit if desired.

7.5 Select the sample units to be inspected. The number of sample units to be inspected may vary from all of the sample units in the section, a number of sample units that provides a 95 % confidence level, or a lesser number.

7.5.1 All sample units in the section may be inspected to determine the average PCI of the section. This is usually

AIRFIELD ASPHALT PAVEMENT CONDITION SURVEY DATA SHEET FOR SAMPLE UNIT	SKETCH:
BRANCH__________ SECTION________ SAMPLE UNIT________ SURVEYED BY_________ DATE________ SAMPLE AREA________	

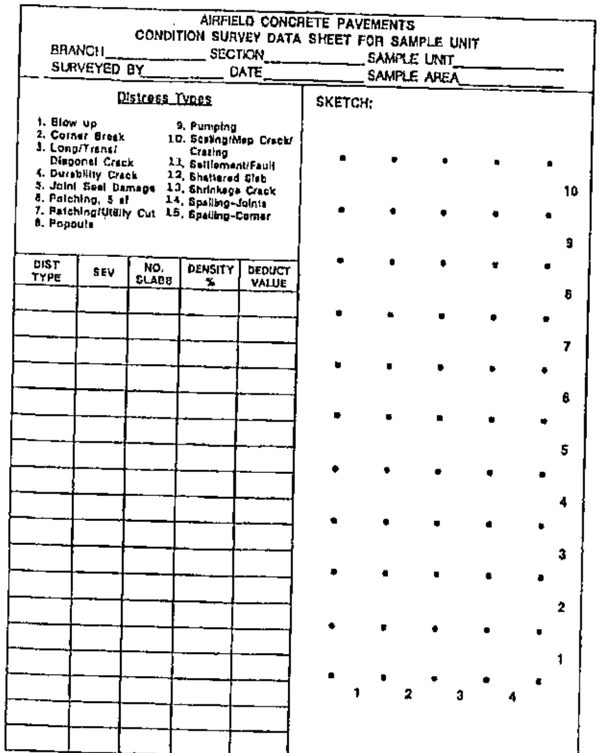
1. Alligator Cracking	5. Depression	9. Oil Spillage	13. Rutting
2. Bleeding	6. Jet Blast	10. Patching	14. Shoving from PCC
3. Block Cracking	7. Jt. Reflection (PCC)	11. Polished Aggregate	15. Slippage Cracking
4. Corrugation	8. Long. & Trans. Cracking	12. Raveling/Weathering	16. Swell

DISTRESS SEVERITY	QUANTITY									TOTAL	DENSITY %	DEDUCT VALUE

FIG. 2 Flexible Pavement Condition Survey Data Sheet for Sample Unit

precluded for routine management purposes by available manpower, funds, and time. Total sampling, however, is desirable for project analysis to help estimate maintenance and repair quantities.

7.5.2 The minimum number of sample units (n) that must be surveyed within a given section to obtain a statistically adequate estimate (95 % confidence) of the PCI of the section


AIRFIELD CONCRETE PAVEMENTS
CONDITION SURVEY DATA SHEET FOR SAMPLE UNIT

BRANCH________ SECTION________ SAMPLE UNIT________
SURVEYED BY________ DATE________ SAMPLE AREA________

Distress Types

1. Blow up
2. Corner Break
3. Long/Trans/ Diagonal Crack
4. Durability Crack
5. Joint Seal Damage
6. Patching, 5 sf
7. Patching/Utility Cut
8. Popouts
9. Pumping
10. Scaling/Map Crack/ Crazing
11. Settlement/Fault
12. Shattered Slab
13. Shrinkage Crack
14. Spalling-Joints
15. Spalling-Corner

DIST TYPE	SEV	NO. SLABS	DENSITY %	DEDUCT VALUE

FIG. 3 Jointed Rigid Pavement Condition Survey Data Sheet for Sample Unit

is calculated using the following formula and rounding n to the next highest whole number (**1**).

$$n = \frac{Ns^2}{\left(\left(\frac{e^2}{4}\right)(N-1) + s^2\right)} \tag{1}$$

where:

e = acceptable error in estimating the section PCI. Commonly, e = ±5 PCI points,
s = standard deviation of the PCI from one sample unit to another within the section. When performing the initial inspection, the standard deviation is assumed to be ten for AC pavements and 15 for PCC pavements. This assumption should be checked as described below after PCI values are determined. For subsequent inspections the standard deviation from the preceding inspection should be used to determine n, and
N = total number of sample units in the section.

7.5.2.1 If obtaining the 95 % confidence level is critical, the adequacy of the number of sample units surveyed must be confirmed. The number of sample units was estimated based on an assumed standard deviation. Calculate the actual standard deviation(s) as follows (**1**):

$$s = \sqrt{\sum_{i=1}^{n} \frac{(PCI_i - PCI_f)^2}{(n-1)}} \tag{2}$$

where:

PCI_i = PCI of surveyed sample unit i,
PCI_f = mean PCI of surveyed sample units, and
n = total number of sample units surveyed.

7.5.2.2 Calculate the revised minimum number of sample units (Eq 1) to be surveyed using the calculated standard deviation (Eq 2). If the revised number of sample units to be surveyed is greater than the number of sample units already surveyed, select and survey additional random sample units. These sample units should be evenly spaced across the section. Repeat the process of checking the revised number of sample units and surveying additional random sample units until the total number of sample units surveyed equals or exceeds the minimum required sample units (n) in Eq 1, using the actual total sample standard deviation).

7.5.3 A lesser sampling rate than the above mentioned 95 % confidence level can be used based on the condition survey objective. As an example, one agency uses the following table for selecting the number of sample units to be inspected for other than project analysis:

Given	Survey
1 to 5 sample units	1 sample unit
6 to 10 sample units	2 sample units
11 to 15 sample units	3 sample units
16 to 40 sample units	4 sample units
over 40 sample units	10 %

7.6 Once the number of sample units to be inspected has been determined, compute the spacing interval of the units using systematic random sampling. Samples are equally spaced throughout the section with the first sample selected at random. The spacing interval (i) of the units to be sampled is calculated by the following formula rounded to the next lowest whole number:

$$i = \frac{N}{n} \tag{3}$$

where:

N = total number of sample units in the section, and
n = number of sample units to be inspected.

The first sample unit to be inspected is selected at random from sample units 1 through i. The sample units within a section that are successive increments of the interval i after the first randomly selected unit are also inspected.

7.7 Additional sample units are only to be inspected when nonrepresentative distresses are observed as defined in 2.1.1. These sample units are selected by the user.

8. Inspection Procedure

8.1 The definitions and guidelines for quantifying distresses for PCI determination are given in Appendix X1 for AC pavements. Other related references (**1, 2, 3, 4, 5, 6, 7, 8**) are also available that discuss distress survey; however, when the material in these references conflict with the definitions included in this test method, the definitions in this test method are used.

8.2 *AC Surfaced Pavement, Including Porous Friction Surfaces*—Individually inspect each sample unit chosen. Sketch the sample unit, including orientation. Record the branch and section number, and number and type of the sample unit (random or additional). Record the sample unit size measured with the hand odometer. Conduct the distress inspection by walking over the sample unit being surveyed, measuring the quantity of each severity level of every distress type present, and recording the data. Distresses must correspond in types and severities to those described in Appendix X1. The method of measurement is included with each distress description. Measurements should be made to ±0.1 ft (30 mm) with the hand odometer. Summarize each distress type and severity level in either square feet or linear feet (square metres or linear metres), depending on the type of distress. Repeat this procedure for each sample unit to be inspected. A blank "Flexible Pavement Condition Survey Data Sheet for Sample Unit" is included in Appendix X5.

8.3 *PCC Pavements*—Individually inspect each sample unit chosen. Sketch the sample unit showing the location of the slabs. Record the sample unit size, branch and section number, number and type of the sample unit (random or additional), the number of slabs in the sample unit, and the slab size measured with the hand odometer. Perform the inspection by walking over each slab of the sample unit being surveyed and recording all distresses existing in the slab along with their severity level. The distress types and severities must correspond with those described in Appendix X2. Summarize the distress types, their severity levels, and the number of slabs in the sample unit containing each type and severity level. Repeat this procedure for each sample unit to be inspected. A blank "Jointed Rigid Pavement Condition Survey Data Sheet for Sample Unit" is included in Appendix X5.

9. Calculation of PCI for AC Pavement, Including Porous Friction Surfaces

9.1 Add up the total quantity of each distress type at each severity level, and record them in the "Total Severities" section. For example, Fig. 4 shows four entries for the Distress Type 8, "Longitudinal and Transverse Cracking:" 9M, 10L, 20L, and 15L. The distress at each severity level is summed and entered in the "Total Severity" section as 45 ft (14 m) of low severity, and 9 ft (3 m) of medium severity "Longitudinal and Transverse Cracking." The units for the quantities may be

AIRFIELD ASPHALT PAVEMENT CONDITION SURVEY DATA SHEET FOR SAMPLE UNIT

SKETCH:

BRANCH________ SECTION________ SAMPLE UNIT________
SURVEYED BY________ DATE________ SAMPLE AREA 5000 S.F.

1. Alligator Cracking
2. Bleeding
3. Block Cracking
4. Corrugation
5. Depression
6. Jet Blast
7. Jt. Reflection (PCC)
8. Long. & Trans. Cracking
9. Oil Spillage
10. Patching
11. Polished Aggregate
12. Raveling/Weathering
13. Rutting
14. Shoving from PCC
15. Slippage Cracking
16. Swell

DISTRESS SEVERITY	QUANTITY									TOTAL	DENSITY %	DEDUCT VALUE
8L	10	20	15							45	0.90	4.8
8M	9									9	0.18	4.9
1L	50									50	1.00	21.0
13L	200	175								375	7.50	27.0
13M	25									25	0.50	20.0
5L	15									15	0.30	2.0
5M	20									20	0.40	9.0
10L	50									50	1.00	4.0

FIG. 4 Example of a Flexible Pavement Condition Survey Data Sheet

either in square feet (square metres), linear feet (metres), or number of occurrences, depending on the distress type.

9.2 Divide the total quantity of each distress type at each severity level from 9.1 by the total area of the sample unit and multiply by 100 to obtain the percent density of each distress type and severity.

9.3 Determine the deduct value (DV) for each distress type and severity level combination from the distress deduct value curves in Appendix X3.

9.4 Determine the maximum corrected deduct value (CDV):

9.4.1 If none or only one individual DV is greater than five, the total value is used in place of the maximum CDV in determining PCI; otherwise, maximum CDV must be determined using the procedure described in this section. The procedure for determining maximum CDV from individual DVs is identical for both AC and PCC pavement types.

9.5 *PCI Calculation:*

9.5.1 If none or only one individual DV is greater than five, use the total DV in place of the maximum CDV in determining PCI; otherwise use the following procedure to determine Max CDV:

9.5.1.1 Determine m, the maximum allowable number of distresses, as follows:

$$m = 1 + (9/95)(100 - HDV) \leq 10 \tag{4}$$

$$m = 1 + (9/95)(100 - 27) = 7.92 \tag{5}$$

$$\text{HDV} = \text{highest individual DV} \tag{6}$$

9.5.1.2 Enter m largest DVs on Line 1 of the following table, including the fraction obtained by multiplying the last DV by the fractional portion of m. If less than m DVs are available, enter all of the DVs.

9.5.1.3 Sum the DVs and enter it under "Total". Count the number of DVs greater than 5.0 and enter it under "q".

9.5.1.4 Look up the appropriate correction curve (AC or PCC) with "Total" and "q" to determine CDV.

9.5.1.5 Copy DVs on current line to the next line, changing the smallest DV greater than five to five. Repeat 9.5.1.3 and 9.5.1.4 until "q" = 1.

9.5.1.6 Maximum CDV is the largest value in the "CDV" column.

9.5.2 List the individual DVs in descending order. For example in Fig. 4 this will be: 27.0, 21.0, 20.0, 9.0, 4.9, 4.8, 4.0, and 2.0.

9.5.3 Determine the allowable number of deducts, m, from Fig. 5, or using the following formulas:

$$m = 1 + (9/95)(100 - HDV) \tag{7}$$

where:

m = allowable number of deducts including fractions (must be less than or equal to ten), and
HDV = highest individual DV.

For the example in Fig. 4:

$$m = 1 + (9/95)(100 - 27.0) = 7.92 \tag{8}$$

9.5.4 The number of individual DVs is reduced to the m largest DVs, including the fractional part. For example, for the values in Fig. 4, the values are: 27.0, 21.0, 20.0, 9.0, 4.9, 4.8, 4.0, and 1.8 (the 1.8 was obtained by multiplying 2.0 by (7.92 − 7 = 0.92)). If less than m DVs are available, all of the DVs are used.

9.5.5 Determine maximum CDV iteratively as follows: (see Fig. 6):

9.5.5.1 Determine the total DV by summing individual DVs. The total DV is obtained by adding the individual DVs in 9.5.4, that is 92.5.

9.5.5.2 Determine q; q is the number of deducts with a value greater than 5.0. For the example in Fig. 4, q = 4.

9.5.5.3 Determine the CDV from q and total DV determined in 9.5.5.1 and 9.5.5.2 by looking up the appropriate correction curve for AC pavements in Fig. X3.19 in Appendix X3.

9.5.5.4 Reduce the smallest individual DV greater than 5.0 to 5.0 and repeat 9.5.5.1-9.5.5.4 until q = 1.

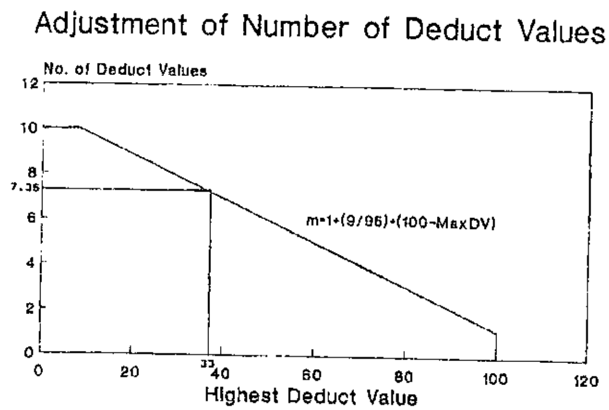


FIG. 5 Adjustment of Number of DVs

#	Deduct Values										Total	q	CDV
1	27.0	21.0	20.0	9.0	4.9	4.8	4.0	1.8			92.5	4	50.0
2	27.0	21.0	20.0	5.0	4.9	4.8	4.0	1.8			38.5	3	56.0
3	27.0	21.0	5.0	5.0	4.9	4.8	4.0	1.8			75.5	2	51.0
4	27.0	5.0	5.0	5.0	4.9	4.8	4.0	1.8			57.5	1	57.5
5													
6													
7													
8													
9													
10													

Max CDV = 57.5

PCI = 100 − Max CDV = 41.5

RATING = FAIR

FIG. 6 Calculation of Corrected PCI Value—Flexible Pavement

9.5.5.5 Maximum CDV is the largest of the CDVs determined in 9.5.5.1-9.5.5.4.

9.6 Calculate PCI by subtracting the maximum CDV from 100 (PCI = 100 - max CDV).

9.7 Fig. 6 shows a summary of PCI calculation for the example AC pavement data in Fig. 4. A blank PCI calculation form is included in Appendix X5.

10. Calculation of PCI for PCC Pavement

10.1 For each unique combination of distress type and severity level, add up the total number of slabs in which they occur. For example in Fig. 7, there are two slabs containing low-severity corner break.

10.2 Divide the number of slabs from 10.1 by the total number of slabs in the sample unit and multiply by 100 to obtain the percent density of each distress type and severity combination.

10.3 *PCI Calculation*:

10.3.1 If none or only one individual DV is greater than five, use the total DV in place of the maximum CDV in determining PCI; otherwise use the following procedure to determine max CDV:

10.3.1.1 Determine m, the maximum allowable number of distresses, as follows:

$$m = 1 + (9/95)(100 - HDV) \leq 10 \tag{9}$$

$$m = 1 + (9/95)(100 - 32.0) = 7.44 \tag{10}$$

$$\text{HDV} = \text{highest individual DV} \tag{11}$$

10.3.1.2 Enter m largest DVs on Line 1 of the following table, including the fraction obtained by multiplying the last DV by the fractional portion of m. If less than m DVs are available, enter all of the DVs.

10.3.1.3 Sum the DVs and enter it under "Total". Count the number of DVs greater than 5.0 and enter it under "q".

10.3.1.4 Look up the appropriate correction curve (AC or PCC) with "Total" and "q" to determine CDV.

10.3.1.5 Copy DVs on current line to the next line, changing the smallest DV greater than five to five. Repeat 10.3.1.3 and 10.3.1.4 until "q" = 1.

10.3.1.6 Maximum CDV is the largest value in the "CDV" column.

10.4 Determine the DVs for each distress type severity level combination using the corresponding deduct curve in Appendix X4.

10.5 Determine PCI by following the procedures in 9.5 and 9.6, using the correction curve for PCC pavements (see Fig. X4.16) in place of the correction curve for AC pavements in 9.5.5.3.

10.6 Fig. 8 shows a summary of PCI calculation for the example PCC pavement distress data in Fig. 7.

11. Determination of Section PCI

11.1 If all surveyed sample units are selected randomly, then the PCI of the section (PCI_s) is calculated as the area weighted PCI of the randomly surveyed sample units ($\overline{PCI_r}$) using Eq 12:

$$PCI_S = \overline{PCI_r} = \frac{\sum_{i=1}^{n} (PCI_{ri} \cdot A_{ri})}{\sum_{i=1}^{n} A_{ri}} \tag{12}$$

AIRFIELD CONCRETE PAVEMENTS
CONDITION SURVEY DATA SHEET FOR SAMPLE UNIT

BRANCH_____ SECTION_____ SAMPLE UNIT_____

SURVEYED BY LMB DATE 18 JAN 92 SAMPLE AREA 12.5' × 25'

Distress Types

1. Blow up
2. Corner Break
3. Long/Trans/Diagonal Crack
4. Durability Crack
5. Joint Seal Damage
6. Patching, ≤ 5 sf
7. Patching/Utility Cut
8. Popouts
9. Pumping
10. Scaling/Map Crack/Crazing
11. Settlement/Fault
12. Shattered Slab
13. Shrinkage Crack
14. Spalling-Joints
15. Spalling-Corner

DIST TYPE	SEV	NO. SLABS	DENSITY %	DEDUCT VALUE
5	H	20	100	12.0
2	L	2	10	8.0
2	M	1	5	9.0
3	L	3	15	11.0
3	M	5	25	32.0
15	L	3	15	6.0
14	L	2	10	3.0
12	L	1	5	10.0

SKETCH:

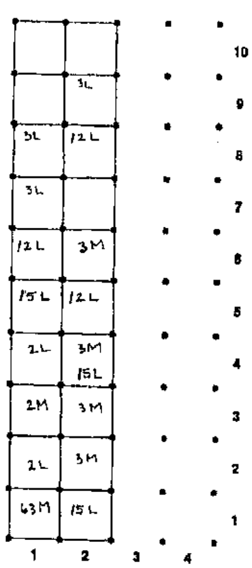


FIG. 7 Example of a Jointed Rigid Pavement Condition Survey Data Sheet

#	Deduct Values										Total	q	CDV
1	32.0	12.0	11.0	10.0	9.0	8.0	6.0	1.3			89.3	7	56.0
2	32.0	12.0	11.0	10.0	9.0	8.0	5.0	1.3			88.3	6	58.0
3	32.0	12.0	11.0	10.0	9.0	5.0	5.0	1.3			85.3	5	58.0
4	32.0	12.0	11.0	10.0	5.0	5.0	5.0	1.3			81.3	4	58.0
5	32.0	12.0	11.0	5.0	5.0	5.0	5.0	1.3			76.3	3	57.0
6	32.0	12.0	5.0	5.0	5.0	5.0	5.0	1.3			70.3	2	61.0
7	32.0	5.0	5.0	5.0	5.0	5.0	5.0	1.3			63.3	1	63.3
8													
9													
10													

Max CDV = 63.3

PCI = 100 – Max CDV = 36.7

RATING = Poor

FIG. 8 Calculation of Corrected PCI Value—Jointed Rigid Pavement where:

$\overline{PCI_r}$ = area weighted PCI of randomly surveyed sample units.

PCI_{ri} = PCI of random sample unit i,

A_{ri} = area of random sample unit i,

n = number of random sample units surveyed.

If additional sample units, as defined in 2.1.1, are surveyed, the area weighted PCI of the surveyed additional units ($\overline{PCI_a}$) is calculated using Eq 13. The PCI of the pavement section is calculated using Eq 14.

$$\overline{PCI_a} = \frac{\sum_{i=1}^{m}(PCI_{ai} \cdot A_{ai})}{\sum_{i=1}^{m} A_{ai}} \quad (13)$$

$$PCI_s = \frac{\overline{PCI_r}(A - \sum_{i=1}^{m} A_{ai}) + \overline{PCI_a}(\sum_{i=1}^{m} A_{ai})}{A} \quad (14)$$

$\overline{PCI_a}$ = area weighted PCI of additional sample units,

PCI_{ai} = PCI of additional sample unit i,

A_{ai} = area of additional sample unit i,

A = area of section,

m = number of additional sample units surveyed, and

PCI_s = area weighted PCI of the pavement section.

11.2 Determine the overall condition rating of the section by using the section PCI and the condition rating scale in Fig. 1.

12. Report

12.1 Develop a summary report for each section. The summary lists section location, size, total number of sample units, the sample units inspected, the PCIs obtained, the average PCI for the section, and the section condition rating.

13. Precision and Bias

13.1 *Precision*—At this time, no precision estimate has been obtained from statistically designed tests. This statement is subject to change in the next five years (see Note 1).

13.2 *Bias*—No statement concerning the bias of the test method can be established at this time.

NOTE 1—Using this test method, inspectors should identify distress types accurately 95 % of the time. Linear measurements should be considered accurate when they are within 10 % if remeasured, and area measurements should be considered accurate when they are within 20 % if remeasured.

APPENDIXES

(Nonmandatory Information)

X1. PAVEMENT CONDITION INDEX (PCI) AC AIRFIELDS

NOTE X1.1—The sections in this appendix are arranged in the following order:

	Section
Alligator Cracking	X1.2
Bleeding	X1.3
Block Cracking	X1.4
Corrugation	X1.5
Depression	X1.6
Jet-Blast Erosion	X1.7
Joint Reflection Cracking	X1.8
Longitudinal and Transverse Cracking	X1.9
Oil Spillage	X1.10
Patching and Utility Cut Patching	X1.11
Polished Aggregate	X1.12
Raveling and Weathering	X1.13
Rutting	X1.14
Shoving	X1.15
Slippage Cracking	X1.16
Swell	X1.17

X1.1 Distresses in Asphalt Pavement—Sixteen distress types for AC pavements are listed alphabetically. During the field condition surveys and the validation of the PCI, several questions were often asked regarding the identification and measurement of some of the distresses. The answers to most of these questions are included under the section "How To Measure", for each distress. For convenience, however, the items that are frequently referenced are listed as follows:

X1.1.1 Spalling as used in this test method is the further breaking of pavement or loss of materials around cracks or joints.

X1.1.2 A crack filler is in satisfactory condition if it is intact. An intact filler prevents water and incompressibles from entering the crack.

X1.1.3 If a crack does not have the same severity level along its entire length, each portion of the crack having a different severity level should be recorded separately. If however, the different levels of severity in a portion of a crack cannot be easily divided, that portion should be rated at the highest severity level present.

X1.1.4 If "alligator cracking" and "rutting" occur in the same area, each is recorded at its respective severity level.

X1.1.5 If "bleeding" is counted, "polished aggregate" is not counted in the same area.

X1.1.6 "Block cracking" includes all of the "longitudinal and transverse cracking" within the area; however, "joint reflection cracking" is recorded separately.

X1.1.7 Any distress, including cracking, found in a patched area is not recorded; however, its effect on the patch is considered in determining the severity level of the patch.

X1.1.8 A significant amount of polished aggregate should be present before it is counted.

X1.1.9 Conducting a PCI survey immediately after the application of surface treatment is not meaningful, because surface treatments mask existing distresses.

X1.1.10 A surface treatment that is coming off should be counted as "raveling."

X1.1.11 A distress is said to have "foreign object damage" (FOD) potential when surficial material is in a broken or loose state, such that the possibility of ingestion of the material into an engine is present, or the potential for freeing the material due to trafficking is present.

X1.1.12 Sections X1.1.1-X1.1.11 are not intended to be a complete list. To properly measure each distress type, the inspector must be familiar with its individual measurement criteria.

X1.2 *Alligator or Fatigue Cracking:*

X1.2.1 *Description*—Alligator or fatigue cracking is a series of interconnecting cracks caused by fatigue failure of the AC surface under repeated traffic loading. The cracking initiates at the bottom of the AC surface (or stabilized base) where tensile stress and strain are highest under a wheel load. The cracks propagate to the surface initially as a series of parallel cracks. After repeated traffic loading, the cracks connect, forming many-sided, sharp-angled pieces that develop a pattern resembling chicken wire or the skin of an alligator. The pieces are less than 2 ft (0.6 m) on the longest side.

X1.2.2 Alligator cracking occurs only in areas that are subjected to repeated traffic loadings, such as wheel paths. Therefore, it would not occur over an entire area unless the entire area was subjected to traffic loading. (Pattern-type cracking that occurs over an entire area that is not subjected to loading is rated as block cracking, that is, not a load-associated distress.)

X1.2.3 Alligator cracking is considered a major structural distress.

X1.2.4 *Severity Levels:*

X1.2.4.1 *L (Low)*—Fine, longitudinal hairline cracks running parallel to one another with none or only a few interconnecting cracks. The cracks are not spalled (see Figs. X1.1-X1.3).

X1.2.4.2 *M (Medium)*— Further development of light alligator cracking into a pattern or network of cracks that may be lightly spalled. Medium-severity alligator cracking is defined by a well-defined pattern of interconnecting cracks, where all pieces are securely held in place (good aggregate interlock between pieces) (see Figs. X1.4-X1.8).

FIG. X1.1 Low-Severity Alligator Cracking

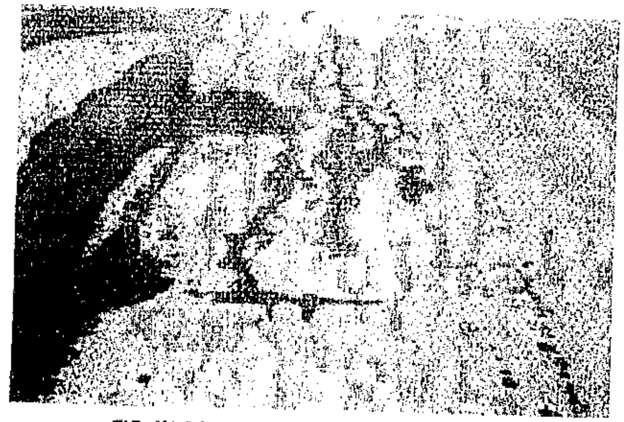


FIG. X1.2 Low-Severity Alligator Cracking

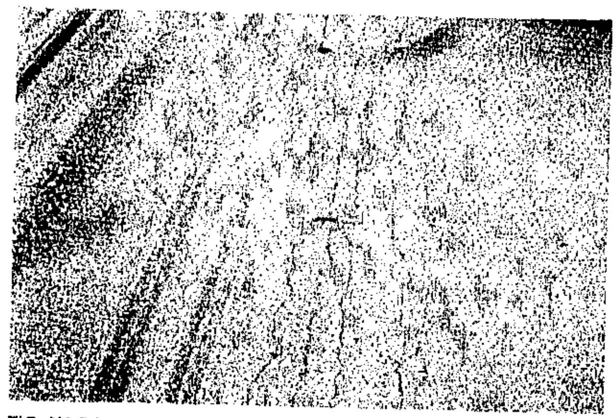


FIG. X1.3 Low-Severity Alligator Cracking, Approaching Medium Severity

FIG. X1.4 Medium-Severity Alligator Cracking (Note the Depression Occurring with the Cracking)

X1.2.4.3 *H (High)*—Network or pattern cracking has progressed so that the pieces are well defined and spalled at the edges; some of the pieces rock under traffic and may cause FOD potential (see Fig. X1.9).

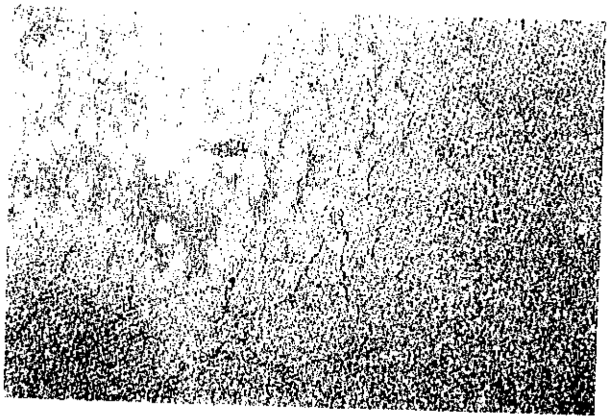


FIG. X1.5 Medium-Severity Alligator Cracking

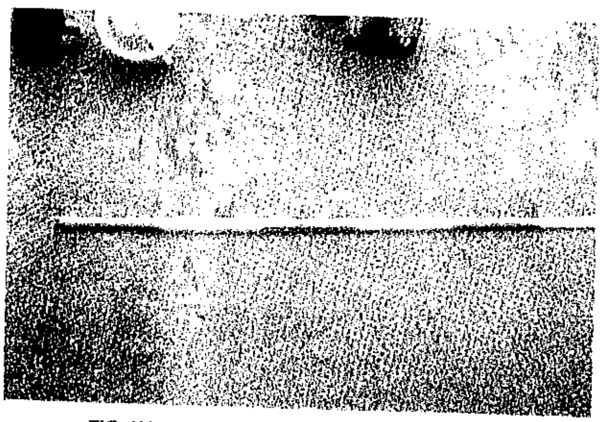


FIG. X1.6 Medium-Severity Alligator Cracking

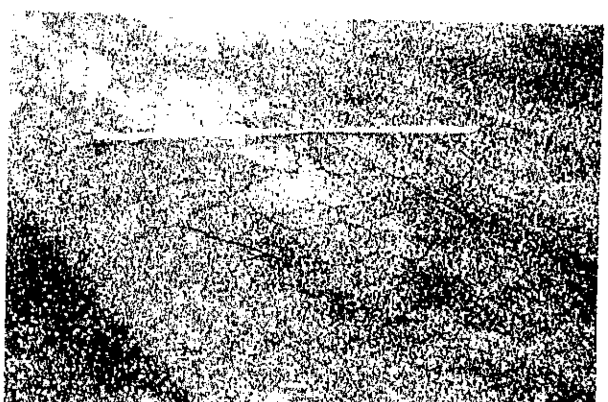


FIG. X1.7 Medium-Severity Alligator Cracking, Approaching High Severity (Example 1)

X1.2.5 *How to Measure*— Alligator cracking is measured in square feet (square metres) of surface area. The major difficulty in measuring this type of distress is that many times two or three levels of severity exist within one distressed area. If these portions can be easily distinguished from one another, they

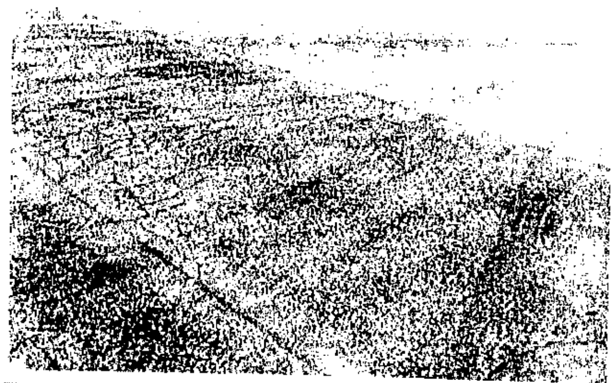


FIG. X1.8 Medium-Severity Alligator Cracking, Approaching High Severity (Example 2)

FIG. X1.9 High-Severity Alligator Cracking

should be measured and recorded separately. However, if the different levels of severity cannot be easily divided, the entire area should be rated at the highest severity level present. If alligator cracking and rutting occur in the same area, each is recorded separately as its respective severity level.

X1.3 *Bleeding:*

X1.3.1 *Description*—Bleeding is a film of bituminous material on the pavement surface that creates a shiny, glass-like, reflecting surface that usually becomes quite sticky. Bleeding is caused by excessive amounts of asphaltic cement or tars in the mix or low-air void content, or both. It occurs when asphalt fills the voids of the mix during hot weather and then expands out onto the surface of the pavement. Since the bleeding process is not reversible during cold weather, asphalt or tar will accumulate on the surface.

X1.3.2 *Severity Levels*—No degrees of severity are defined (see Fig. X1.10 and Fig. X1.11).

X1.3.3 *How to Measure*—Bleeding is measured in square feet (square metres) of surface area.

X1.4 *Block Cracking:*

X1.4.1 *Description*—Block cracks are interconnected cracks that divide the pavement into approximately rectangular

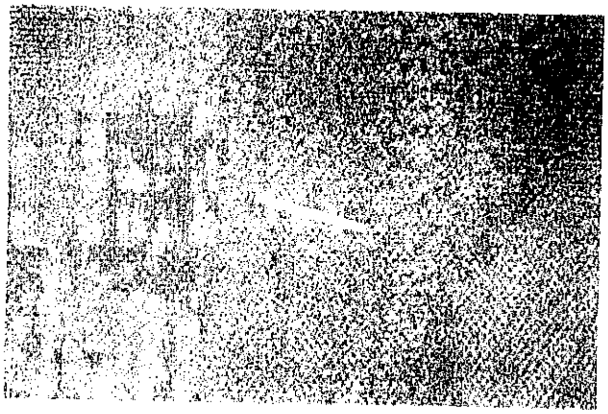


FIG. X1.10 Bleeding

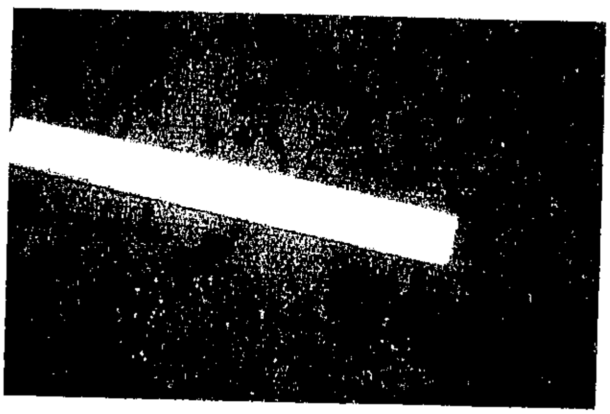


FIG. X1.11 Close-Up of Fig. X1.10

FIG. X1.12 Low-Severity Block Cracking

pieces. The blocks may range in size from approximately 1 by 1 ft to 10 by 10 ft (0.3 by 0.3 m to 3 by 3 m). Block cracking is caused mainly by shrinkage of the AC and daily temperature cycling (that results in daily stress/strain cycling). It is not load associated. The occurrence of block cracking usually indicates that the asphalt has hardened significantly. Block cracking normally occurs over a large portion of pavement area, but sometimes will occur only in nontraffic areas. This type of distress differs from alligator cracking in that the alligator cracks form smaller, many-sided pieces with sharp angles. Also unlike block cracks, alligator cracks are caused by repeated traffic loadings and are, therefore, located only in traffic areas (that is, wheel paths).

X1.4.2 *Severity Levels*:

X1.4.2.1 *L*—Blocks are defined by cracks that are nonspalled (sides of the crack are vertical) or lightly spalled, causing no FOD potential. Nonfilled cracks have ¼ in. (6 mm) or less mean width and filled cracks have filler in satisfactory condition (see Figs. X1.12-X1.15).

X1.4.2.2 *M*—Blocks are defined by either: filled or nonfilled cracks that are moderately spalled (some FOD potential); nonfilled cracks that are not spalled or have only minor spalling (some FOD potential), but have a mean width greater than approximately ¼ in. (6 mm); or filled cracks greater than ¼ in. that are not spalled or have only minor spalling (some FOD potential), but have filler in unsatisfactory condition (see Fig. X1.16 and Fig. X1.17).

X1.4.2.3 *H*—Blocks are well defined by cracks that are severely spalled, causing a definite FOD potential (see Figs. X1.18-X1.20).

X1.4.3 *How to Measure*—Block cracking is measured in square feet (square metres) of surface area, and usually occurs at one severity level in a given pavement section; however, any areas of the pavement section having distinctly different levels of severity should be measured and recorded separately. For asphalt pavements, not including AC over PCC, if block cracking is recorded, no longitudinal and transverse cracking should be recorded in the same area. For asphalt overlay over concrete, block cracking, joint reflection cracking, and longitudinal and transverse cracking reflected from old concrete should all be recorded separately.

X1.5 *Corrugation:*

X1.5.1 *Description*—Corrugation is a series of closely spaced ridges and valleys (ripples) occurring at fairly regular intervals (usually less than 5 ft) (1.5 m) along the pavement. The ridges are perpendicular to the traffic direction. Traffic

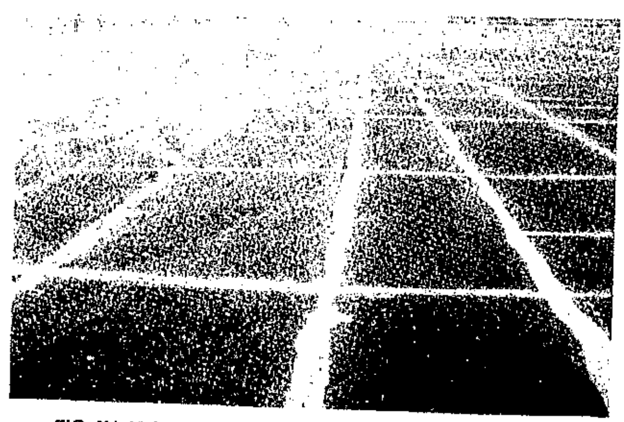


FIG. X1.13 Low-Severity Block Cracking, Filled Cracks

FIG. X1.14 Low-Severity Block Cracking, Filled Cracks

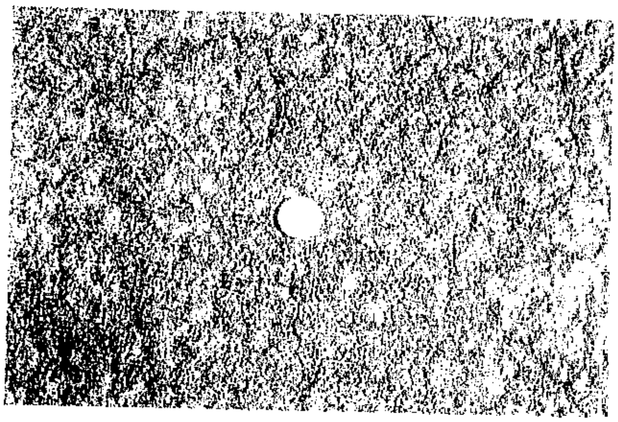


FIG. X1.15 Low-Severity Block Cracking, Small Blocks Defined by Hairline Cracks

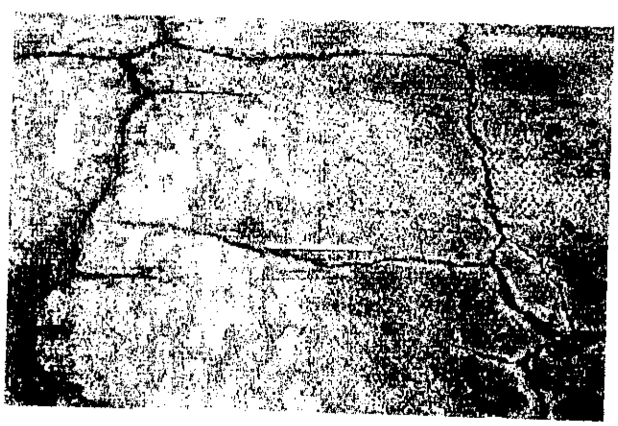


FIG. X1.16 Medium-Severity Block Cracking

action combined with an unstable pavement surface or base usually causes this type of distress.

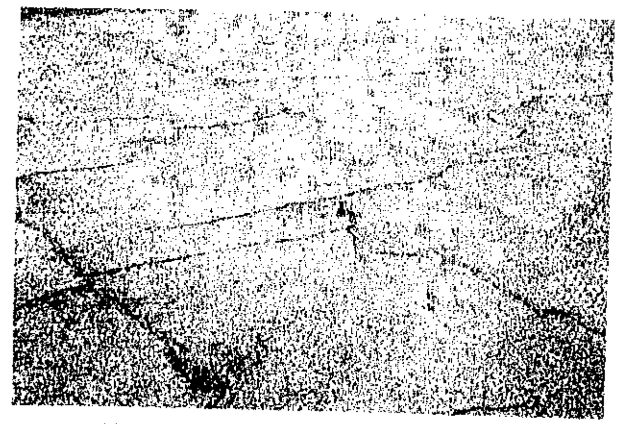


FIG. X1.17 Medium-Severity Block Cracking

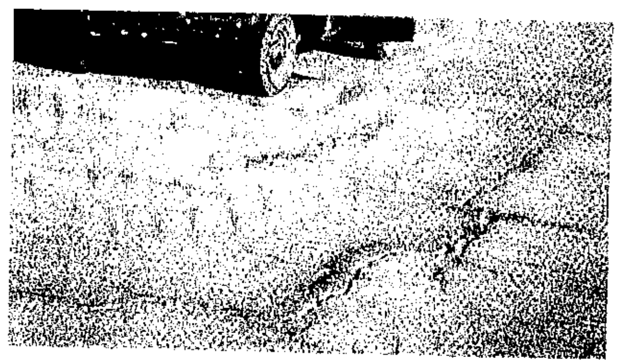


FIG. X1.18 High-Severity Block Cracking

FIG. X1.19 High-Severity Block Cracking

X1.5.2 *Severity Levels*:

X1.5.2.1 *L*—Corrugations are minor and do not significantly affect ride quality (see measurement criteria below) (see Fig. X1.21).

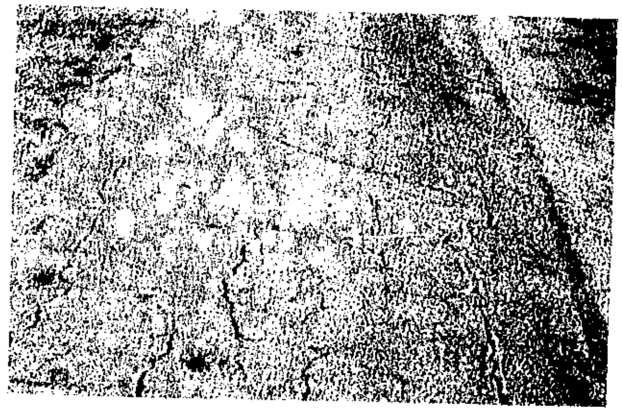


FIG. X1.20 High-Severity Block Cracking

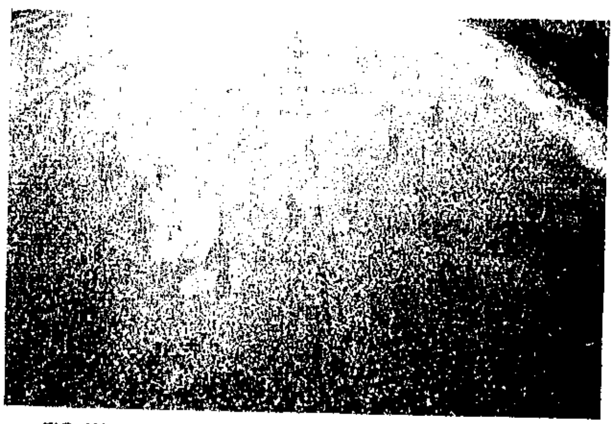


FIG. X1.21 Low-Severity Corrugation in the Foreground, Changing to Medium and High in the Background

X1.5.2.2 *M*—Corrugations are noticeable and significantly affect ride quality (see measurement criteria below) (see Fig. X1.22).

X1.5.2.3 *H*—Corrugations are easily noticed and severely affect ride quality (see measurement criteria below) (see Fig. X1.23).

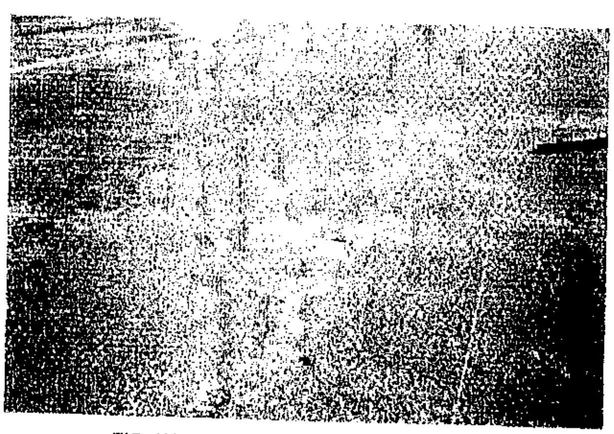


FIG. X1.22 Medium-Severity Corrugation

FIG. X1.23 High-Severity Corrugation

X1.5.3 *How to Measure*—Corrugation is measured in square feet (square metres) of surface area. The mean elevation difference between the ridges and valleys of the corrugations indicates the level of severity. To determine the mean elevation difference, a 10-ft (3-m) straightedge should be placed perpendicular to the corrugations so that the depth of the valleys can be measured in inches (millimetres). The mean depth is calculated from five such measurements.

Severity	Runways and High-Speed Taxiways	Taxiways and Aprons
L	< ¼ in. (6 mm)	< ½ in. (13 mm)
M	¼ to ½ in. (6 to 13 mm)	½ to 1 in. (13 to 25 mm)
H	> ½ in. (13 mm)	> 1 in. (25 mm)

X1.6 *Depression:*

X1.6.1 *Description*—Depressions are localized pavement surface areas having elevations slightly lower than those of the surrounding pavement. In many instances, light depressions are not noticeable until after a rain, when ponding water creates "birdbath" areas; but the depressions can also be located without rain because of stains created by ponding of water. Depressions can be caused by settlement of the foundation soil or can be built during construction. Depressions cause roughness and, when filled with water of sufficient depth, could cause hydroplaning of aircraft.

X1.6.2 *Severity Levels:*

X1.6.2.1 *L*—Depression can be observed or located by stained areas, only slightly affects pavement riding quality, and may cause hydroplaning potential on runways (see measurement criteria below) (see Fig. X1.24).

X1.6.2.2 *M*—The depression can be observed, moderately affects pavement riding quality, and causes hydroplaning potential on runways (see measurement criteria below) (see Fig. X1.25 and Fig. X1.26).

X1.6.2.3 *H*—The depression can be readily observed, severely affects pavement riding quality, and causes definite hydroplaning potential (see measurement criteria below) (see Fig. X1.27).

X1.6.3 *How to Measure*—Depressions are measured in square feet (square metres) of surface area. The maximum depth of the depression determines the level of severity. This

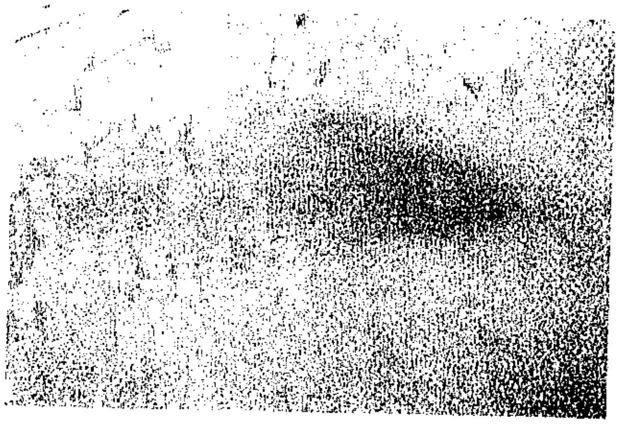


FIG. X1.24 Low-Severity Depression

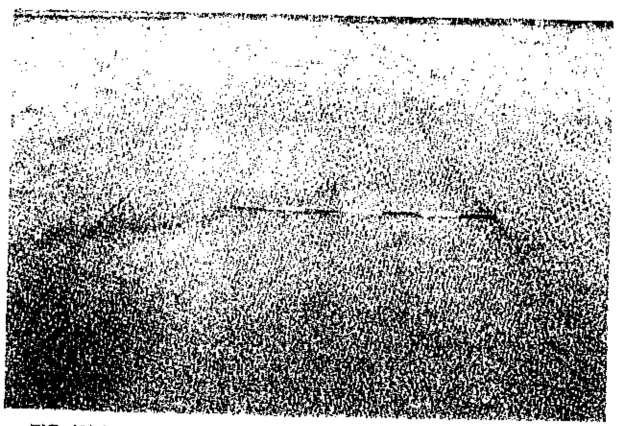


FIG. X1.25 Medium-Severity Depression (1½ in. (37.5 mm))

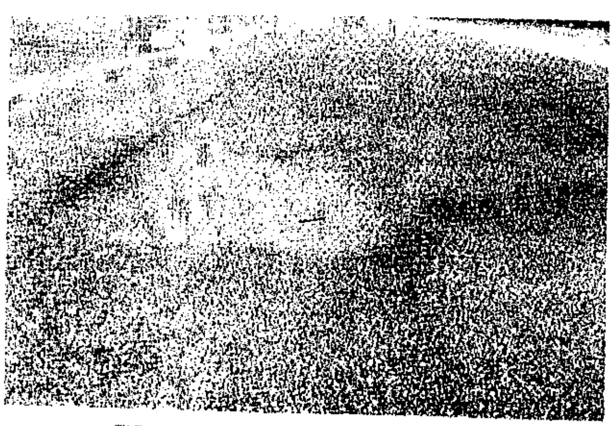


FIG. X1.26 Medium-Severity Depression

depth can be measured by placing a 10-ft (3-m) straightedge across the depressed area and measuring the maximum depth in inches (millimetres). Depressions larger than 10 ft (3 m) across must be measured by using a stringline:

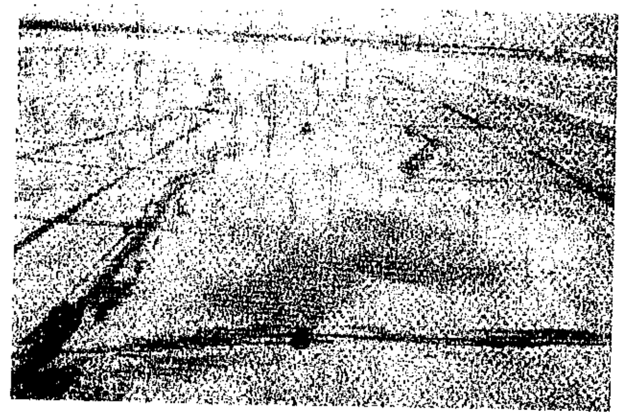


FIG. X1.27 High-Severity Depression (2 in. (50 mm))

Severity	Maximum Depth of Depression	
	Runways and High-Speed Taxiways	Taxiways and Aprons
L	⅛ to ½ in. (3 to 13 mm)	½ to 1 in. (13 to 25 mm)
M	½ to 1 in. (13 to 25 mm)	1 to 2 in. (25 to 51 mm)
H	> 1 in. (> 25 mm)	> 2 in. (> 51 mm)

X1.7 *Jet-Blast Erosion:*

X1.7.1 *Description*—Jet-blast erosion causes darkened areas on the pavement surface where bituminous binder has been burned or carbonized. Localized burned areas may vary in depth up to approximately ½ in. (13 mm).

X1.7.2 *Severity Levels*—No degrees of severity are defined. It is sufficient to indicate that jet-blast erosion exists (see Fig. X1.28 and Fig. X1.29).

X1.7.3 *How to Measure*—Jet-blast erosion is measured in square feet (square metres) of surface area.

X1.8 *Joint Reflection Cracking From PCC (Longitudinal and Transverse):*

X1.8.1 *Description*—This distress occurs only on pavements having an asphalt or tar surface over a PCC slab. This category does not include reflection cracking from any other

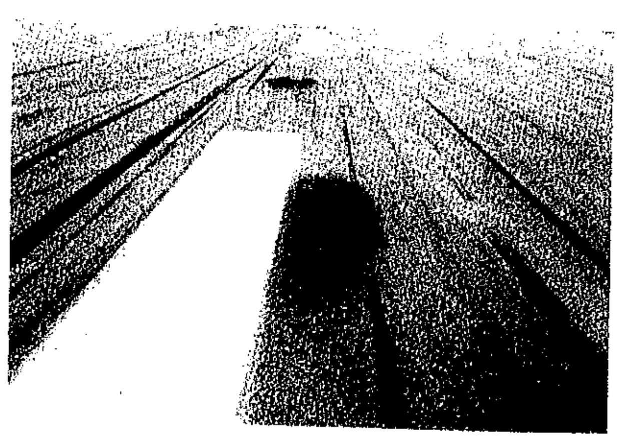


FIG. X1.28 Jet-Blast Erosion

FIG. X1.29 Jet-Blast Erosion

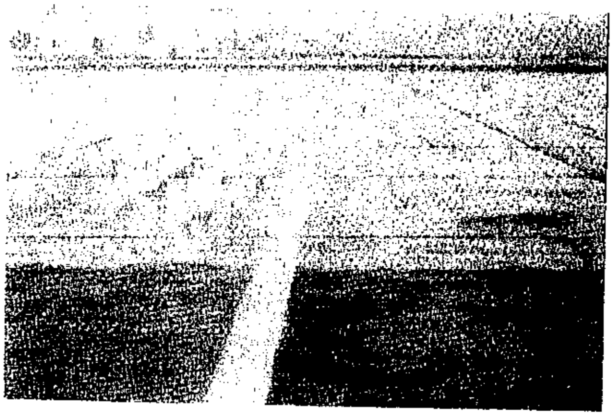


FIG. X1.30 Low-Severity Joint Reflection Cracking

FIG. X1.31 Low-Severity Joint Reflection Cracking, Filled Crack

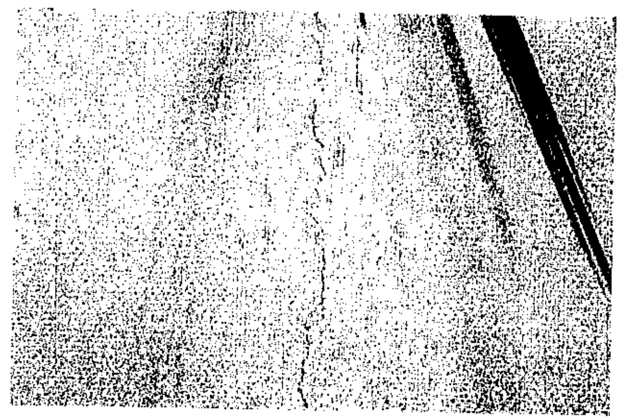


FIG. X1.32 Low-Severity Joint Reflection Cracking, Nonfilled Crack

type of base (that is, cement stabilized, lime stabilized). Such cracks are listed as longitudinal and transverse cracks. Joint reflection cracking is caused mainly by movement of the PCC slab beneath the AC surface because of thermal and moisture changes; it is not load-related. However, traffic loading may cause a breakdown of the AC near the crack, resulting in spalling and FOD potential. If the pavement is fragmented along a crack, the crack is said to be spalled. A knowledge of slab dimensions beneath the AC surface will help to identify these cracks.

X1.8.2 *Severity Levels*:

X1.8.2.1 *L*—Cracks have only light spalling (little or no FOD potential) or no spalling, and can be filled or nonfilled. If nonfilled, the cracks have a mean width of ¼ in. (6 mm) or less; filled cracks are of any width, but their filler material is in satisfactory condition (see Figs. X1.30-X1.32).

X1.8.2.2 *M*—One of the following conditions exists: cracks are moderately spalled (some FOD potential) and can be either filled or nonfilled of any width; filled cracks are not spalled or are lightly spalled, but filler is in unsatisfactory condition; nonfilled cracks are not spalled or are only lightly spalled, but the mean crack width is greater than ¼ in. (6 mm); or light random cracking exists near the crack or at the corners of intersecting cracks (see Figs. X1.33-X1.35).

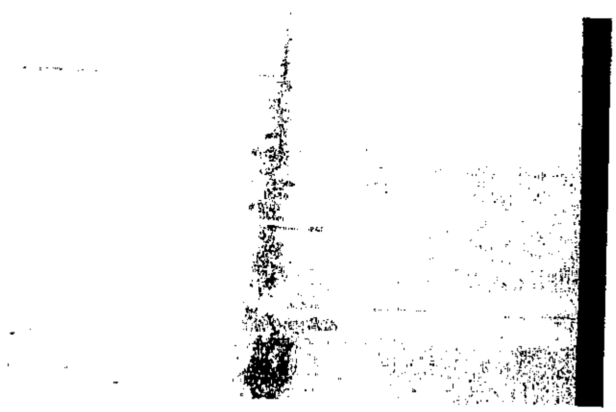


FIG. X1.33 Medium-Severity Joint Reflection Cracking

X1.8.2.3 *H*—Cracks are severely spalled with pieces loose or missing causing definite FOD potential. Cracks can be either filled or nonfilled of any width (see Fig. X1.36).

X1.8.3 *How to Measure*—Joint reflection cracking is measured in linear feet (metres). The length and severity level of

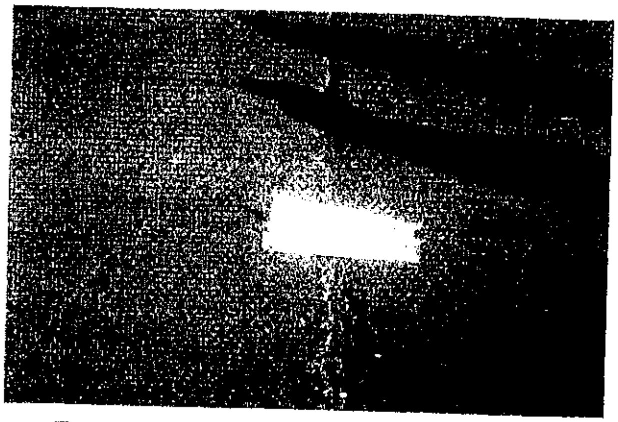


FIG. X1.34 Medium-Severity Joint Reflection Cracking

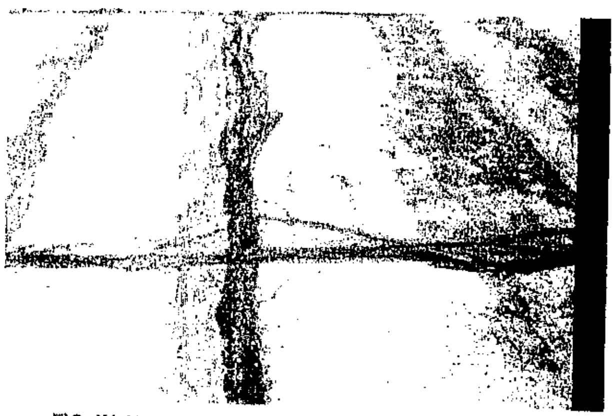


FIG. X1.35 Medium-Severity Joint Reflection Cracking

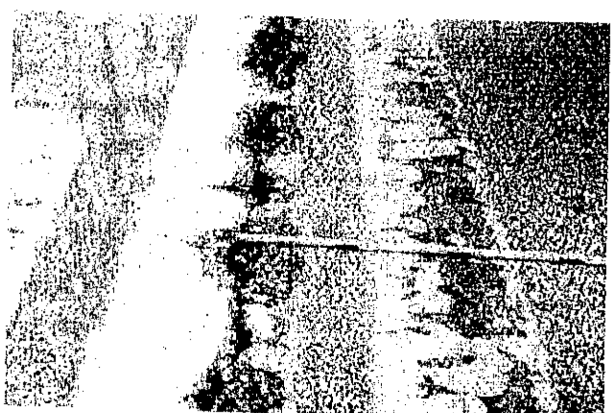


FIG. X1.36 High-Severity Joint Reflection Cracking

each crack should be identified and recorded. If the crack does not have the same severity level along its entire length, each portion should be recorded separately. For example, a crack that is 50 ft (15 m) long may have 10 ft (3 m) of a high severity, 20 ft (6 m) of a medium severity, and 20 ft (6 m) of a light severity. These would all be recorded separately. If the different levels of severity in a portion of a crack cannot be easily divided, that portion should be rated at the highest severity present.

X1.9 *Longitudinal and Transverse Cracking (Non-PCC Joint Reflective):*

X1.9.1 *Description*—Longitudinal cracks are parallel to the pavement's center line or laydown direction. They may be caused by (*1*) a poorly constructed paving lane joint, (*2*) shrinkage of the AC surface due to low temperatures or hardening of the asphalt, or (*3*) a reflective crack caused by cracks beneath the surface course, including cracks in PCC slabs (but not at PCC joints). Transverse cracks extend across the pavement at approximately right angles to the pavement's center line or direction of laydown. They may be caused by (*2*) or (*3*). These types of cracks are not usually load associated. If the pavement is fragmented along a crack, the crack is said to be spalled.

X1.9.2 *Severity Levels*:

X1.9.2.1 *L*—Cracks have only light spalling (little or no FOD potential) or no spalling, and can be filled or nonfilled. If nonfilled, the cracks have a mean width of ¼ in. (6 mm) or less; filled cracks are of any width, but their filler material is in satisfactory condition (see Fig. X1.37 and Fig. X1.38).

X1.9.2.2 *M*—One of the following conditions exists: (*1*) cracks are moderately spalled (some FOD potential) and can be either filled or nonfilled of any width; (*2*) filled cracks are not spalled or are lightly spalled, but filler is in unsatisfactory condition; (*3*) nonfilled cracks are not spalled or are only lightly spalled, but the mean crack width is greater than ¼ in. (6 mm), or (*4*) light random cracking exists near the crack or at the corners of intersecting cracks (see Figs. X1.39-X1.41).

X1.9.2.3 *H*—Cracks are severely spalled and pieces are loose or missing causing definite FOD potential. Cracks can be either filled or nonfilled of any width (see Fig. X1.42).

X1.9.3 *Porous Friction Courses: Severity Levels*:

X1.9.3.1 *L*—Average raveled area around the crack is less than ¼ in. (6 mm) wide (see Fig. X1.43).

X1.9.3.2 *M*—Average raveled area around the crack is between ¼ to 1 in. (6 to 25 mm) wide (see Fig. X1.44).

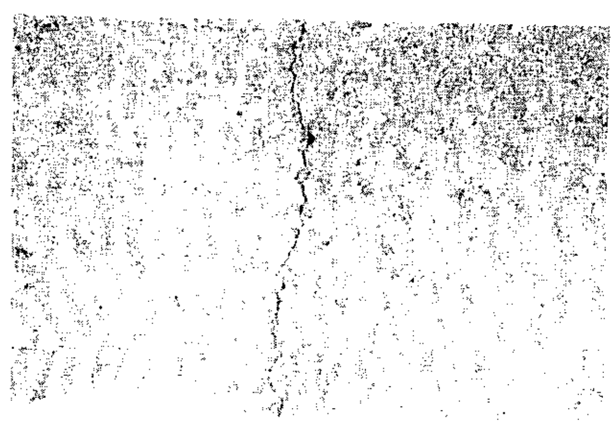


FIG. X1.37 Low-Severity Longitudinal Crack

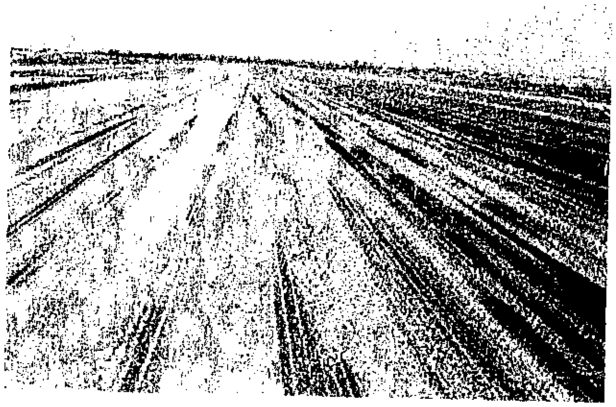


FIG. X1.38 Low-Severity Longitudinal Cracks, Approaching Medium

FIG. X1.39 Medium-Severity Longitudinal Construction Joint Crack

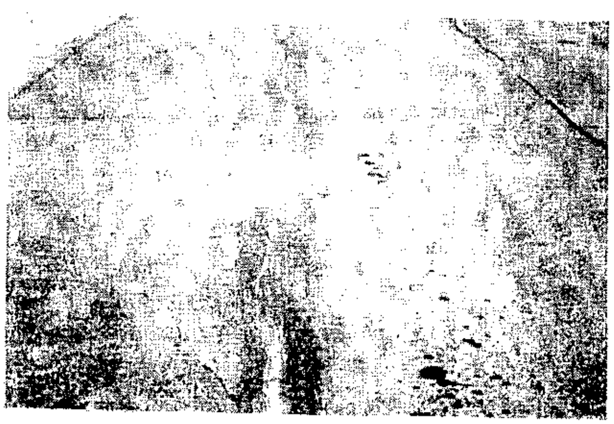


FIG. X1.40 Medium-Severity Longitudinal Crack (Note the Crack is Reflective But Not at the Joint of Slab)

X1.9.3.3 *H*—Average raveled area around the crack is greater than 1 in. (25 mm) wide (see Fig. X1.45).

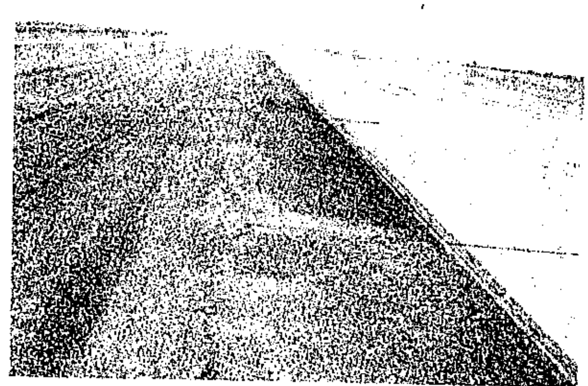


FIG. X1.41 Medium-Severity Longitudinal Crack

FIG. X1.42 High-Severity Longitudinal Crack

X1.9.4 *How to Measure*—Longitudinal and transverse cracks are measured in linear feet (metres). The length and severity of each crack should be identified and recorded. If the crack does not have the same severity level along its entire length, each portion of the crack having a different severity level should be recorded separately. For an example see "Joint Reflection Cracking." If block cracking is recorded, longitudinal and transverse cracking is not recorded in the same area.

X1.10 *Oil Spillage:*

X1.10.1 *Description*—Oil spillage is the deterioration or softening of the pavement surface caused by the spilling of oil, fuel, or other solvents.

X1.10.2 *Severity Levels*—No degrees of severity are defined. It is sufficient to indicate that oil spillage exists (see Fig. X1.46 and Fig. X1.47).

X1.10.3 *How to Measure*—Oil spillage is measured in square feet (square metres) of surface area. A stain is not a distress unless material has been lost or binder has been softened. If hardness is approximately the same as on surrounding pavement, and if no material has been lost, do not record as a distress.

X1.11 *Patching and Utility Cut Patch:*

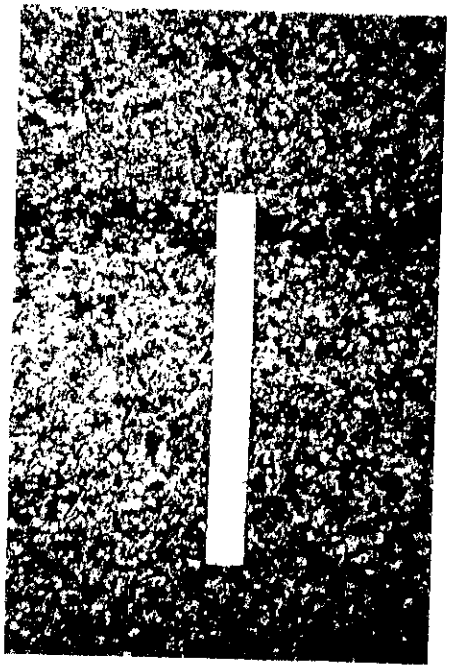


FIG. X1.43 Low-Severity Crack in Porous Friction Course

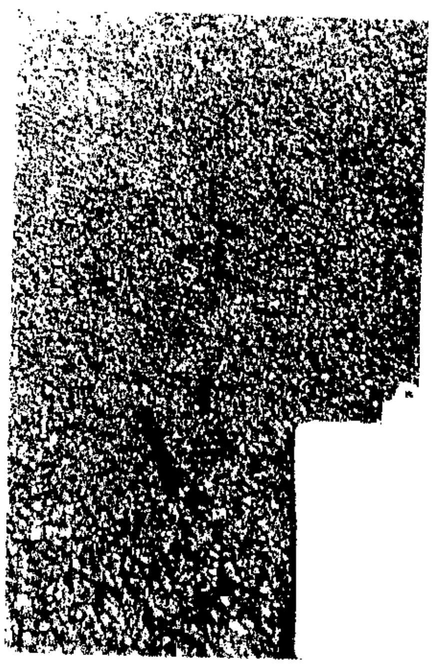


FIG. X1.44 Medium-Severity Crack in Porous Friction Course

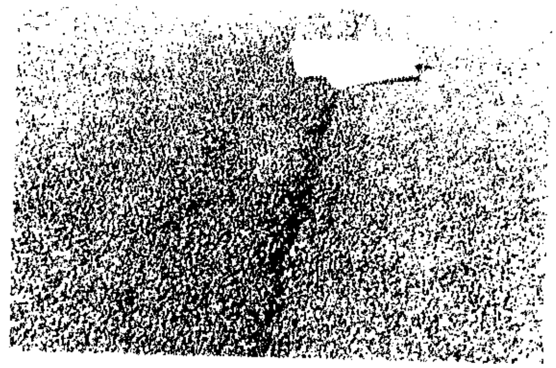


FIG. X1.45 High-Severity Crack in Porous Friction Course

FIG. X1.46 Oil Spillage

FIG. X1.47 Oil Spillage

X1.11.1 *Description*— A patch is considered a defect, no matter how well it is performing.

X1.11.2 *Severity Levels*:

X1.11.2.1 *L*—Patch is in good condition and is performing satisfactorily (see Figs. X1.48-X1.50).

X1.11.2.2 *M*—Patch is somewhat deteriorated and affects ride quality to some extent. Moderate amount of distress is present within the patch or has FOD potential, or both. (see Fig. X1.51).

X1.11.2.3 *H*—Patch is badly deteriorated and affects ride quality significantly or has high FOD potential. Patch soon needs replacement.

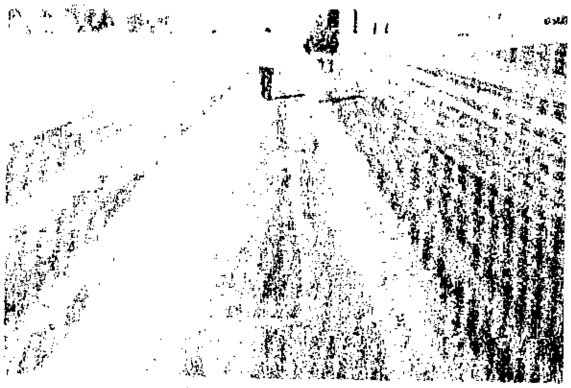


FIG. X1.48 Low-Severity Patch

FIG. X1.49 Low-Severity Patch

FIG. X1.50 Low-Severity Patch with Medium-Severity Portion

FIG. X1.51 Medium-Severity Patch

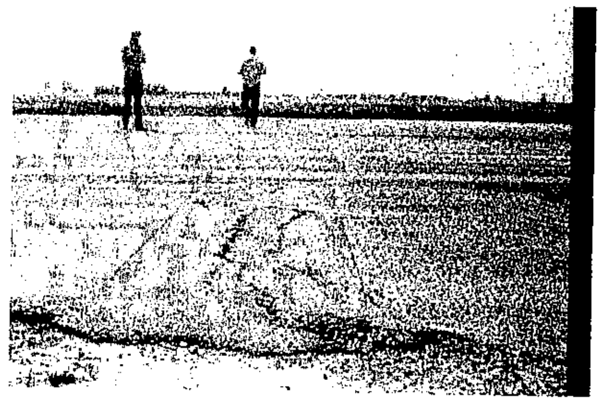


FIG. X1.52 High-Severity Patch

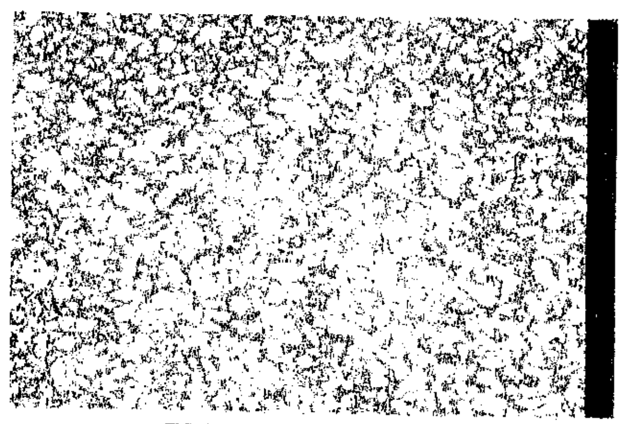


FIG. X1.53 Polished Aggregate

X1.11.3 *Porous Friction Courses*—The use of dense-graded AC patches in porous friction surfaces causes a water damming effect at the patch which contributes to differential skid resistance of the surface. Low-severity dense-graded patches should be rated as medium severity due to the differential friction problem. Medium- and high-severity patches are rated the same as above.

X1.11.4 *How to Measure*:

X1.11.4.1 Patching is measured in square feet (square metres) of surface area. However, if a single patch has areas of differing severity levels, these areas should be measured and recorded separately. For example, a 25-ft^2 (2.5-m^2) patch may have 10 ft^2 (1 m^2) of medium severity and 15 ft^2 (1.5 m^2) of low severity. These areas should be recorded separately. Any distress found in a patched area will not be recorded; however, its effect on the patch will be considered when determining the patch's severity level.

X1.11.4.2 A very large patch, (area > 2500 ft^2 (230 m^2)) or feathered-edge pavement, may qualify as an additional sample unit or as a separate section.

X1.12 *Polished Aggregate:*

X1.12.1 *Description*—Aggregate polishing is caused by repeated traffic applications. Polished aggregate is present when close examination of a pavement reveals that the portion of aggregate extending above the asphalt is either very small, or there are no rough or angular aggregate particles to provide good skid resistance.

X1.12.2 *Severity Levels*—No degrees of severity are defined. However, the degree of polishing should be clearly evident in the sample unit, in that the aggregate surface should be smooth to the touch.

X1.12.3 *How to Measure*—Polished aggregate is measured in square feet (square metres) of surface area. Polished aggregate areas should be compared visually with adjacent nontraffic areas. If the surface texture is substantially the same in both traffic and nontraffic areas, polished aggregate should not be counted.

X1.13 *Raveling and Weathering:*

X1.13.1 *Description*—Raveling and weathering are the wearing away of the pavement surface caused by the dislodging of aggregate particles and loss of asphalt or tar binder. They may indicate that the asphalt binder has hardened significantly. As used in this test method, fine aggregate refers to aggregate with a largest dimension less than ⅜ in. (10 mm), and coarse aggregate refers to aggregate with a smallest dimension greater than or equal to ⅜ in. (10 mm).

X1.13.2 *Dense Mix: Severity Levels*

X1.13.2.1 *L*—(*1*) The surface is in generally good condition, but fine aggregate and binder have worn away exposing the coarse aggregate. The coarse aggregate, however, is still firmly embedded in the mix (see Fig. X1.54). (*2*) In a square yard representative sample, the number of coarse aggregate pieces missing is between 5 and 20 (see Fig. X1.55). (*3*) In a square yard representative sample, brushing one's foot across the surface does not dislodge more than 20 coarse aggregate pieces. If in doubt, three representative square yards should be inspected and the number of missing pieces of coarse aggregate counted.

X1.13.2.2 *M*—(*1*) In a square yard representative sample, the number of coarse aggregate pieces missing is between 21 and 40 (see Fig. X1.56). (*2*) In a square yard representative sample, brushing one's foot across the surface dislodges between 21 and 40 coarse aggregate pieces. If in doubt, three representative square yards should be inspected and the number of missing pieces of aggregate counted.

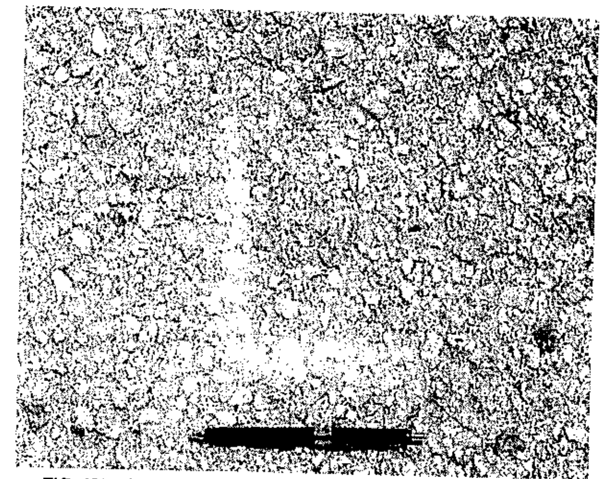


FIG. X1.54 Low-Severity Raveling/Weathering, Dense Mix

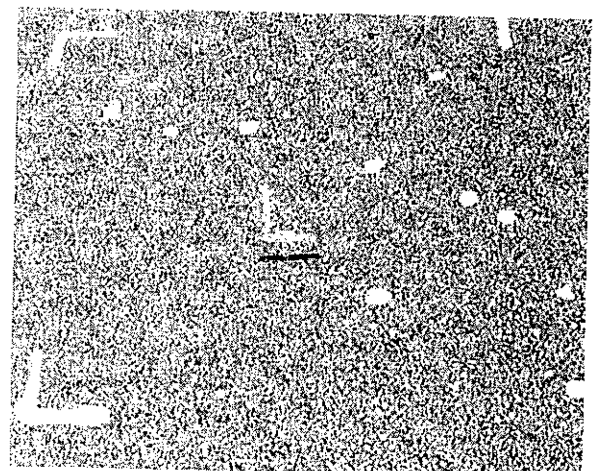


FIG. X1.55 Low-Severity Raveling/Weathering, Dense Mix

X1.13.2.3 *H*—(*1*) In a square yard representative sample, the number of coarse aggregate pieces missing is over 40 (see Fig. X1.57). (*2*) In a square yard representative sample, brushing one's foot across the surface dislodges more than 40 coarse aggregate pieces. If in doubt, three representative square yards should be inspected and the number of missing pieces of aggregate counted.

X1.13.3 *Surface Treatment Over Dense Mix: Severity Levels*

X1.13.3.1 *L*—(*1*) Scaled area is less than 1 %, e.g. less than 3.5 by 3.5 in./yd^2(less than 100 by 100 mm/m^2). (*2*) In case of cold tar where pattern cracking has developed, the tar surface cracks are less than ¼ in. (6 mm) wide (see Fig. X1.58).

X1.13.3.2 *M*—(*1*) Scaled area is between 1 and 10 % (see Fig. X1.57). (2) In case of cold tar where pattern cracking has developed, the tar surface cracks are ¼ in. (6 mm) wide or greater (see Fig. X1.59).

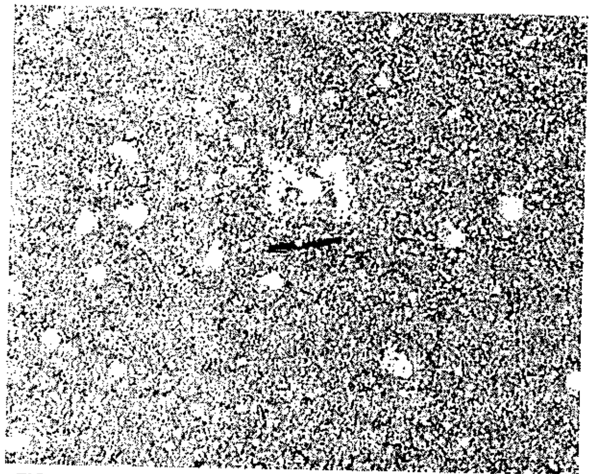


FIG. X1.56 Medium-Severity Raveling/Weathering, Dense Mix

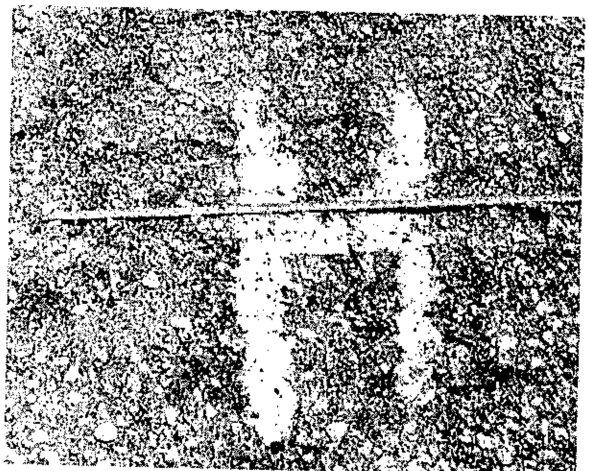


FIG. X1.57 High-Severity Raveling/Weathering, Dense Mix

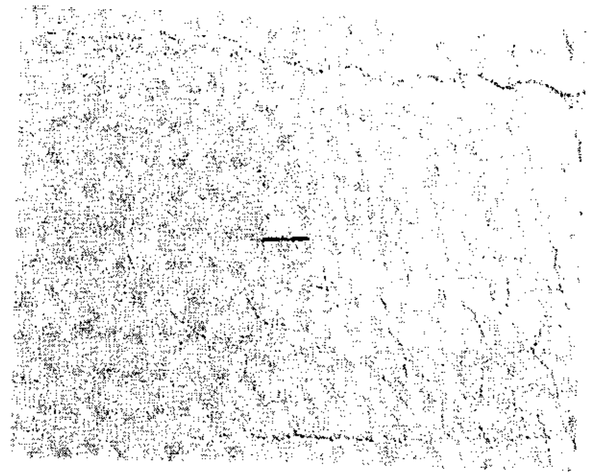


FIG. X1.58 Low-Severity Raveling/Weathering, Cold Tar

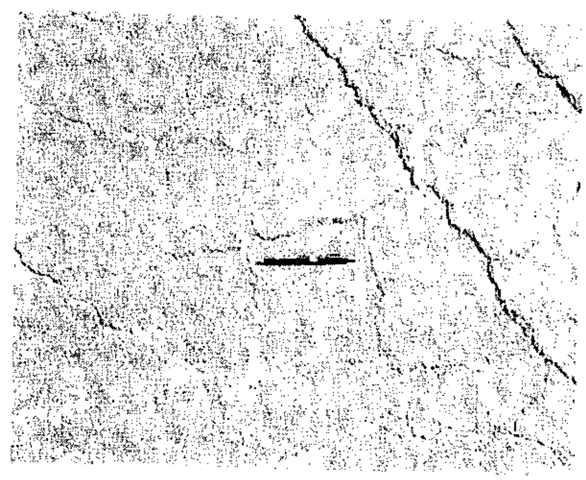


FIG. X1.59 Medium-Severity Raveling/Weathering, Cold Tar

X1.13.3.3 *H*—(*1*) Scaled area is over 10 %, e.g. over 11.5 by 11.5 in./yd^2(over 300 by 300 mm/m^2) (see Fig. X1.58 and Fig. X1.59). (*2*) In case of cold tar, the tar surface is peeling off (see Fig. X1.60).

X1.13.4 *Porous Friction Courses: Severity Levels (see Figs. X1.61-X1.65)*:

X1.13.4.1 *Low Severity*—(*1*) Number of missing small aggregate clusters is between 5 and 20, and little or no FOD potential is present or (*2*) number of missing large aggregate clusters does not exceed 1, and little or no FOD potential is present, or both.

X1.13.4.2 *Medium Severity*—(*1*) Number of missing small aggregate clusters is between 21 and 40, and some FOD potential is present or (*2*) number of missing large aggregate clusters is greater than 1 but less than or equal to 25 % of the one square foot (0.1 square meter) area, and some FOD potential is present, or both.

X1.13.4.3 *High Severity*—(*1*) Number of missing small aggregate clusters is greater than 40, and definite FOD potential is present or (*2*) number of missing large aggregate clusters is greater than 25 % or the square foot area, and definite FOD potential is present, or both.

X1.13.5 *How to Measure*—Raveling and weathering are measured in square feet (square metres) of surface area. The number of missing aggregate pieces should not be counted if they are associated with a crack, instead they will be reflected in the severity of the crack. Mechanical damage caused by equipment, such as snowplows, is counted as high severity.

X1.14 *Rutting:*

X1.14.1 *Description*—A rut is a surface depression in the wheel path. Pavement uplift may occur along the sides of the rut; however, in many instances ruts are noticeable only after a rainfall, when the wheel paths are filled with water. Rutting stems from a permanent deformation in any of the pavement layers or subgrade, usually caused by consolidation or lateral movement of the materials due to traffic loads. Significant rutting can lead to major structural failure of the pavement.

X1.14.2 *Severity Levels*:

FIG. X1.60 High-Severity Raveling/Weathering, Cold Tar

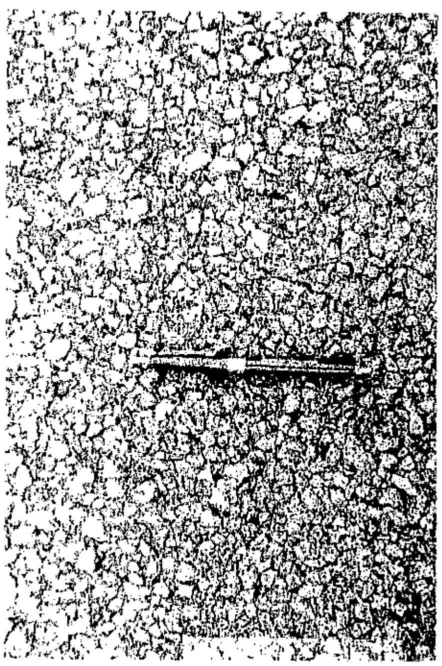


FIG. X1.61 Typical Porous Friction Course Surface with No Raveling/Weathering

Severity	Mean Rut Depth Criteria All Pavement Sections	Figure
L	¼ to ½ in. (< 6 to 13 mm)	Fig. X1.66 and Fig. X1.67
M	> ½ to 1 in. (> 13 to < 25 mm)	Fig. X1.68
H	> 1 in. (> 25 mm)	Fig. X1.69 and Fig. X1.70

X1.14.3 *How to Measure*—Rutting is measured in square feet (square metres) of surface area, and its severity is determined by the mean depth of the rut. To determine the mean depth, a straightedge should be laid across the rut and the depth measured. The mean depth in inches (millimetres) should be computed from measurements taken along the length of the rut. If alligator cracking and rutting occur in the same area, each is recorded at the respective severity level.

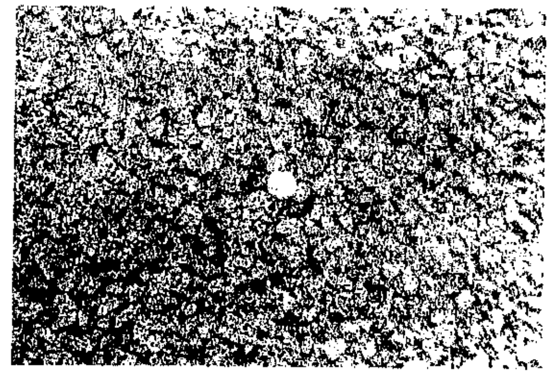


FIG. X1.62 Typical Porous Friction Course Surface with No Raveling/Weathering

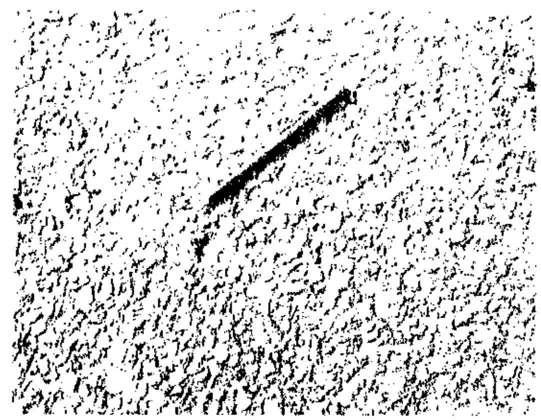


FIG. X1.63 Low-Severity Raveling/Weathering on a Porous Friction Course Surface

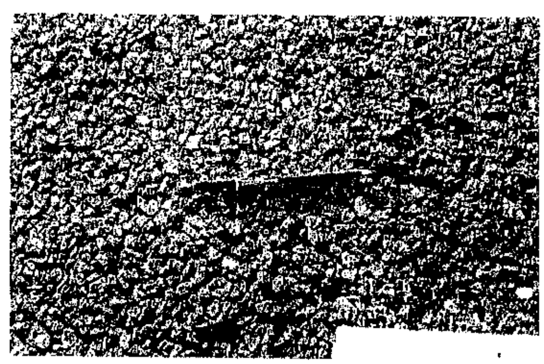


FIG. X1.64 Medium-Severity Raveling/Weathering on a Porous Friction Course Surface

X1.15 *Shoving of Asphalt Pavement by PCC Slabs:*

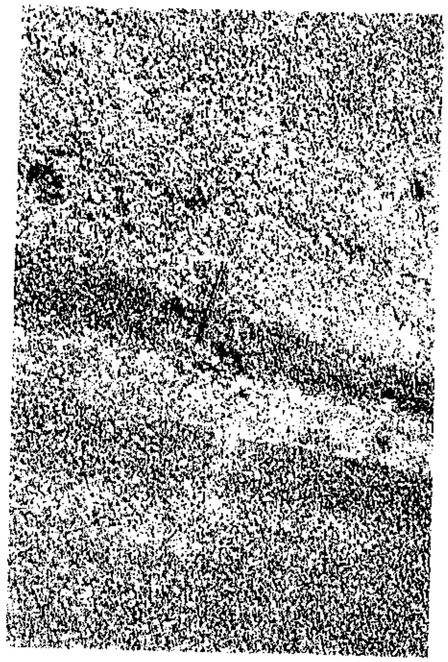


FIG. X1.65 High-Severity Ravelling/Weathering on a Porous Friction Course Surface

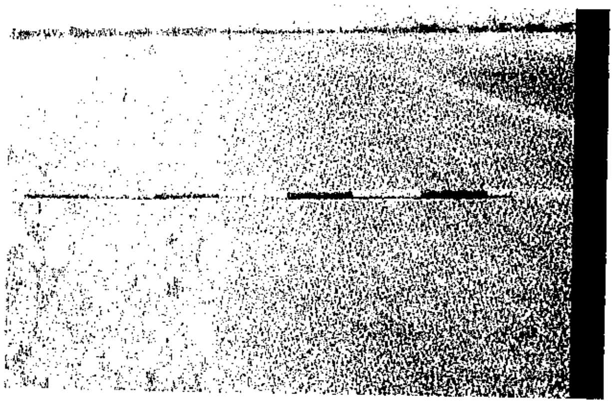


FIG. X1.66 Low-Severity Rutting

X1.15.1 *Description*—PCC pavements occasionally increase in length at ends where they adjoin flexible pavements (commonly referred to as "pavement growth"). This "growth" shoves the asphalt- or tar-surfaced pavements, causing them to swell and crack. The PCC slab "growth" is caused by a gradual opening up of the joints as they are filled with incompressible materials that prevent them from reclosing.

X1.15.2 *Severity Level:*

Severity	Height Differential
L	< ¾ in. (< 20 mm)
M	¾ to 1½ in. (> 20 to 40 mm)
H	> 1½ in. (> 40 mm)

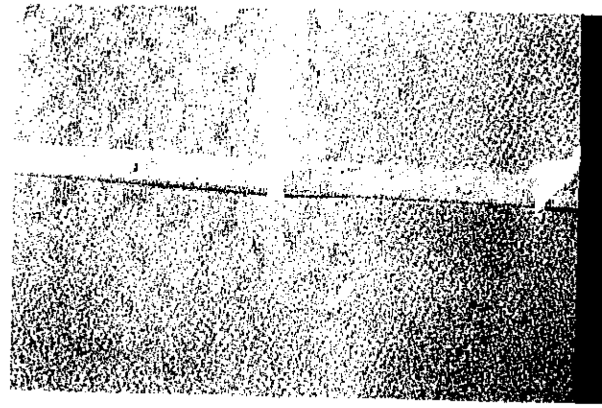


FIG. X1.67 Low-Severity Rutting

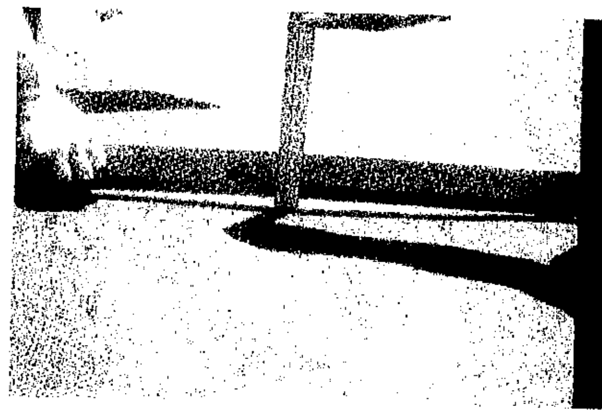


FIG. X1.68 Medium-Severity Rutting

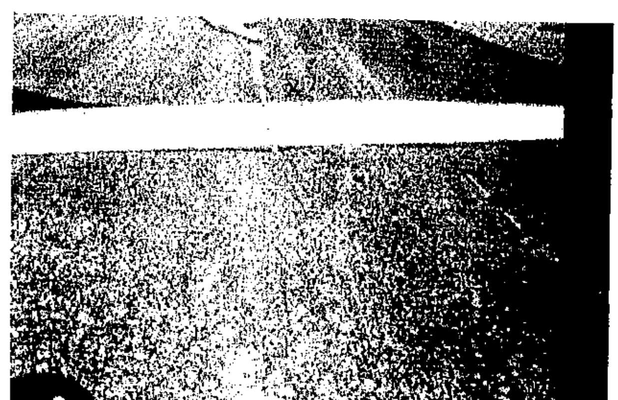


FIG. X1.69 High-Severity Rutting (Note Alligator Cracking Associated With Rutting)

NOTE X1.2—As a guide, the swell table (above) may be used to determine the severity levels of shoving. At the present time, no significant research has been conducted to quantify levels of severity of shoving.

X1.15.2.1 *L*—A slight amount of shoving has occurred and no breakup of the asphalt pavement (see Fig. X1.71).

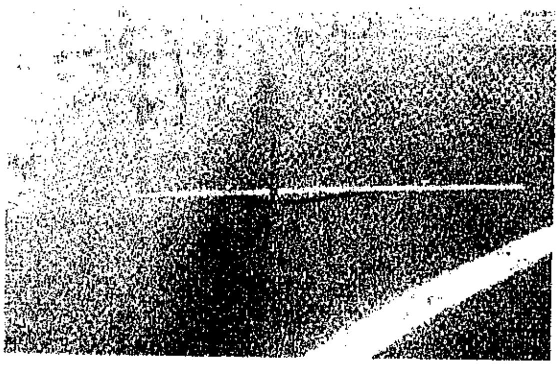


FIG. X1.70 High-Severity Rutting

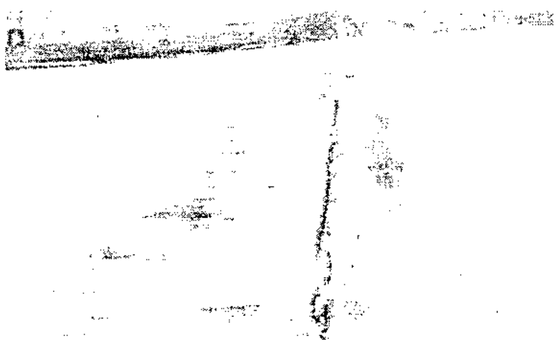


FIG. X1.71 Shove of Low Severity on the Outside and Medium Severity in the Middle

X1.15.2.2 *M*—A significant amount of shoving has occurred, causing moderate roughness and little or no breakup of the asphalt pavement (see Fig. X1.71).

X1.15.2.3 *H*—A large amount of shoving has occurred, causing severe roughness or breakup of the asphalt pavement (see Fig. X1.72).

X1.15.2.4 *How to Measure*—Shoving is measured by determining the area in square feet (square metres) of the swell caused by shoving.

X1.16 *Slippage Cracking:*

X1.16.1 *Description*—Slippage cracks are crescent- or half-moon-shaped cracks having two ends pointed away from the direction of traffic. They are produced when braking or turning wheels cause the pavement surface to slide and deform. This usually occurs when there is a low-strength surface mix or poor bond between the surface and next layer of pavement structure.

X1.16.2 *Severity Levels*—No degrees of severity are defined. It is sufficient to indicate that a slippage crack exists (see Fig. X1.73 and Fig. X1.74).

X1.16.3 *How to Measure*—Slippage cracking is measured in square feet (square metres) of surface area.

FIG. X1.72 High-Severity Shoving

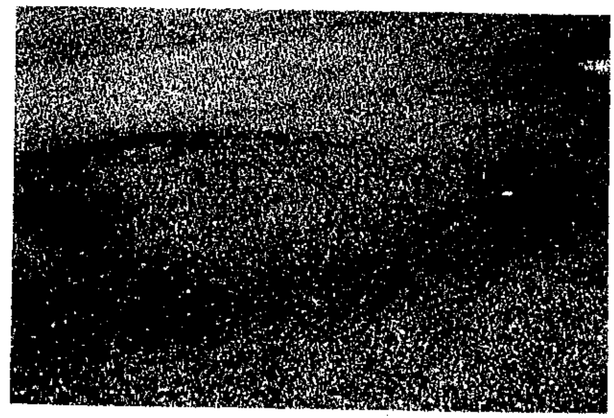


FIG. X1.73 Slippage Cracking

FIG. X1.74 Slippage Cracking

X1.17 *Swell-Distress:*

X1.17.1 *Description*—Swell is characterized by an upward bulge in the pavement's surface. A swell may occur sharply over a small area or as a longer, gradual wave. Either type of swell can be accompanied by surface cracking. A swell is

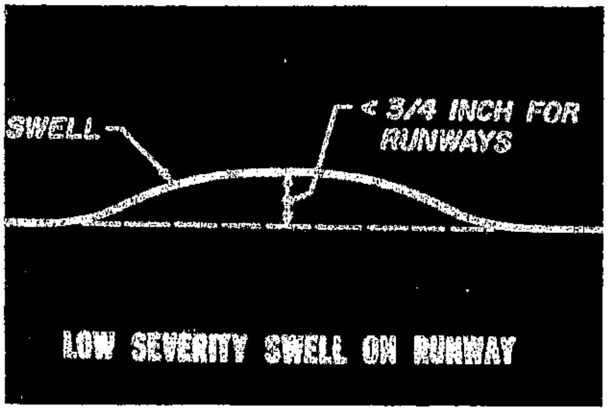


FIG. X1.75 Low-Severity Swell

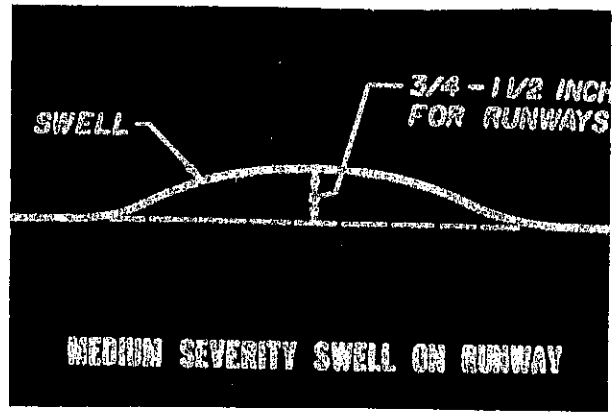


FIG. X1.76 Medium-Severity Swell

usually caused by frost action in the subgrade or by swelling soil, but a small swell can also occur on the surface of an asphalt overlay (over PCC) as a result of a blowup in the PCC slab.

X1.17.2 *Severity Levels*:

X1.17.2.1 *L*—Swell is barely visible and has a minor effect on the pavement's ride quality. (Low-severity swells may not always be observable, but their existence can be confirmed by driving a vehicle over the section. An upward acceleration will occur if the swell is present) (see Fig. X1.75).

X1.17.2.2 *M*—Swell can be observed without difficulty and has a significant effect on the pavement's ride quality (see Fig. X1.76).

X1.17.2.3 *H*—Swell can be readily observed and severely affects the pavement's ride quality (see Fig. X1.77 and Fig. X1.78).

X1.17.3 *How to Measure*:

X1.17.3.1 The surface area of the swell is measured in square feet (square metres). The severity rating should consider the type of pavement section (that is, runway, taxiway, or apron). For example, a swell of sufficient magnitude to cause considerable roughness on a runway at high speeds would be rated as more severe than the same swell located on an apron or taxiway where the normal aircraft operating speeds are much lower.

X1.17.3.2 For short wavelengths, locate the highest point of the swell. Rest at 10-ft (3-m) straightedge on that point so that both ends are equal distance above pavement. Measure this distance to establish severity rating.

FIG. X1.77 High-Severity Swell

X1.17.3.3 The following guidance is provided for runways:

Severity	Height Differential
L	< ¾ in. (20 mm)
M	¾ to 1½ in. (20 to 40 mm)
H	> 1½ in. (40 mm)

Rate severity on high-speed taxiways using measurement criteria provided above. Double the height differential criteria for other taxiways and aprons.

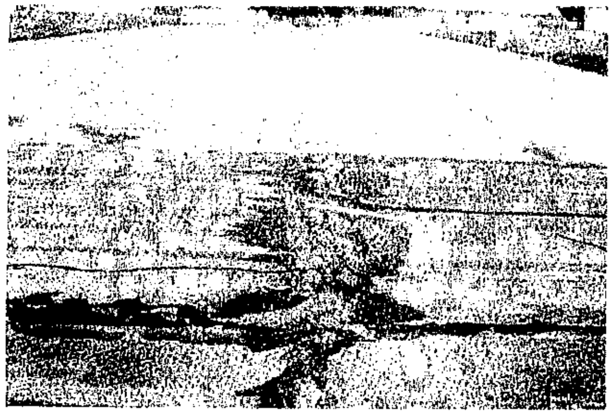


FIG. X1.78 High-Severity Swell

X2. PCI CONCRETE-SURFACED AIRFIELDS

NOTE X2.1—The sections in this appendix are arranged in the following order:

	Section
Distresses in Jointed Concrete Pavement	X2.1
Blowup	X2.2
Corner Break	X2.3
Cracks: Longitudinal, Transverse, and Diagonal	X2.4
Durability ("D") Cracking	X2.5
Joint Seal Damage	X2.6
Patching, Small	X2.7
Patching, Large and Utility Cuts	X2.8
Popouts	X2.9
Pumping	X2.10
Scaling, Map Cracking, Crazing	X2.11
Settlement or Faulting	X2.12
Shattered Slab/Intersecting Cracks	X2.13
Shrinkage Cracks	X2.14
Spalling (Longitudinal and Transverse Joint)	X2.15
Spalling (Corner)	X2.16

X2.1 *Distresses in Jointed Concrete Pavement:*

X2.1.1 Fifteen distress types for jointed concrete pavements are listed alphabetically. The distress definitions apply to both plain and reinforced jointed concrete pavements, with the exception of linear cracking distress, that is defined separately for plain and reinforced jointed concrete pavements.

X2.1.2 During field condition surveys and validation of the PCI, several questions were often asked regarding the identification and counting method of some of the distresses. The answers to most of these questions are included under the section "How to Count" for each distress. For convenience, however, the items that are frequently referenced are listed as follows:

X2.1.2.1 Spalling as used in this test method is the further breaking of the pavement or loss of materials around cracks and joints.

X2.1.2.2 The cracks in reinforced concrete slabs that are less than ⅛ in. (3 mm) wide are counted as "shrinkage cracks." The "shrinkage cracks" should not be counted in determining whether or not the slab is broken into four or more pieces (or "shattered").

X2.1.2.3 Crack widths should be measured between the vertical walls, not from the edge of spalls. Spalling and associated FOD potential are considered in determining the severity level of cracks, but they should not influence the crack width measurements.

X2.1.2.4 A crack filler is in satisfactory condition if it prevents water and incompressibles from entering the crack or joint.

X2.1.2.5 "Joint seal damage" is not counted on a slab-by-slab basis. Instead, the severity level is assigned based on the overall condition of the joint seal in the sample unit.

X2.1.2.6 Do not count a joint as spalled if it can be filled with joint filler.

X2.1.2.7 A premolded joint sealant is in satisfactory condition if it is pliable, firmly against the joint wall, and not extruded.

X2.1.2.8 A fragmented crack is actually two or more cracks in close proximity that meet below the surface forming a single channel to subbase. The multiple cracks are interconnected to form small fragments, or pieces, of pavement.

X2.1.2.9 A crack wider than 3 in. (75 mm) rates at high severity regardless of filler condition.

X2.1.2.10 A spalled or chipped crack edge is defined by secondary cracks, with or without missing pieces, nearly parallel to the primary crack. Individual stones or particles that are dislodged do not constitute spalling.

X2.1.2.11 Little, light, or minor crack edge spalling is defined by secondary cracks typically less than 6 in. (150 mm) long and affecting less than 10 % of the crack length.

X2.1.2.12 Moderate spalling means secondary cracks can be of any length but both ends must intersect the primary crack. Individual pieces wider than 3 in. (75 mm) are not cracked and broken. Some loose particles means loose pieces can be of any length but must be less than 3 in. wide (75 mm) (chips). Missing pieces wider than 3 in. (75 mm) must affect less than 10 % of the crack length.

X2.1.2.13 A distress is said to have FOD potential when surficial material is in a broken or loose state, such that the possibility of ingestion of the material into an engine is present, or the potential for freeing the material due to trafficking is present.

X2.1.3 Sections X2.1.2.1-X2.1.2.13 are not intended to be a complete list. To properly count each distress type, the inspector must be familiar with its individual counting criteria.

X2.2 *Blowup:*

X2.2.1 *Description*—Blowups occur in hot weather, usually at a transverse crack or joint that is not wide enough to permit expansion of the concrete slabs. The insufficient width is usually caused by infiltration of incompressible materials into the joint space. When expansion cannot relieve enough pressure, a localized upward movement of the slab edges (buckling) or shattering will occur in the vicinity of the joint. Blowups can also occur at utility cuts and drainage inlets. This type of distress is almost always repaired immediately because of severe damage potential to aircraft. The main reason blowups are included here is for reference when closed sections are being evaluated for reopening.

X2.2.2 *Severity Levels*:

X2.2.2.1 At the present time, no significant research has been conducted to quantify severity levels for blowups. Future research may provide measurement guidelines:

	Difference in Elevation	
	Runways and High-Speed Taxiways	Aprons and Other Taxiways
L	< ½ in. (< 13 mm)	¼ < 1 in. (6 to 25 mm)
M	½ to 1 in. (13 to 25 mm)	1 to 2 in. (25 to 51 mm)
H	inoperable	inoperable

NOTE X2.2—The elevations are twice the heights used for settlement/faulting. These are preliminary elevations, and subject to change.

X2.2.2.2 *L (Low)*—Buckling or shattering has not rendered the pavement inoperable, and only a slight amount of roughness exists (see Fig. X2.1).

X2.2.2.3 *M (Medium)*—Buckling or shattering has not rendered the pavement inoperable, but a significant amount of roughness exists (see Fig. X2.2).

NOTE 1—This would only be considered low severity if the shattering in the foreground was the only part existing and the foreign material removed.

FIG. X2.1 Low-Severity Blowup

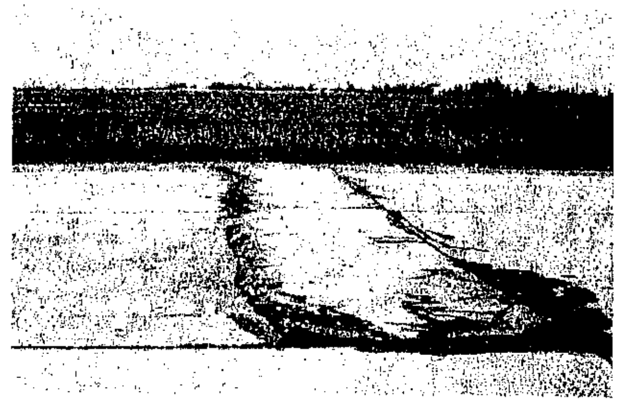


FIG. X2.2 Medium-Severity Blowup

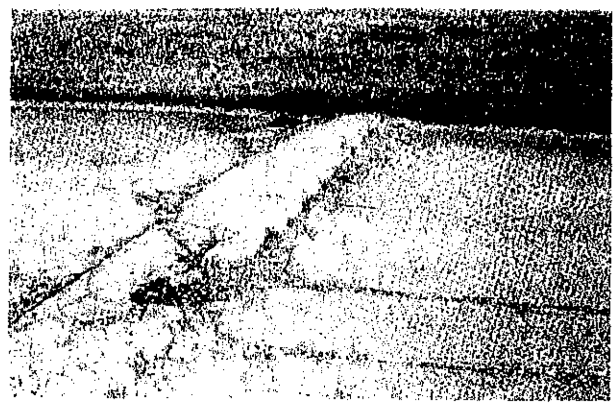


FIG. X2.3 High-Severity Blowup

X2.2.2.4 *H (High)*—Buckling or shattering has rendered the pavement inoperable (see Fig. X2.3).

X2.2.2.5 For the pavement to be considered operational, all foreign material caused by the blowup must have been removed.

X2.2.3 *How to Count*:

X2.2.3.1 A blowup usually occurs at a transverse crack or joint. At a crack, it is counted as being in one slab, but at a joint, two slabs are affected and the distress should be recorded as occurring in two slabs.

X2.2.3.2 Record blowup on a slab only if the distress is evident on that slab. Severity may be different on adjacent slabs. If blowup has been repaired by patching, establish severity by determining the difference in elevation between the two slabs.

X2.3 *Corner Break:*

X2.3.1 *Description*—A corner break is a crack that intersects the joints at a distance less than or equal to one half of the slab length on both sides, measured from the corner of the slab. For example, a slab with dimensions of 25 by 25 ft (7.5 by 7.5 m) that has a crack intersecting the joint 5 ft (1.5 m) from the corner on one side and 17 ft (5 m) on the other side is not considered a corner break; it is a diagonal crack. However, a crack that intersects 7 ft (2 m) on one side and 10 ft (3 m) on the other is considered a corner break. A corner break differs from a corner spall in that the crack extends vertically through the entire slab thickness, while a corner spall intersects the joint at an angle. Load repetition combined with loss of support and curling stresses usually cause corner breaks.

X2.3.2 *Severity Levels:*

X2.3.2.1 *L*—Crack has little or minor spalling (no FOD potential). If nonfilled, it has a mean width less than approximately ⅛ in. (3 mm). A filled crack can be of any width, but the filler material must be in satisfactory condition. The area between the corner break and the joints is not cracked (see Fig. X2.4 and Fig. X2.5).

X2.3.2.2 *M*—One of the following conditions exists: (*1*) filled or nonfilled crack is moderately spalled (some FOD potential); (*2*) a nonfilled crack has a mean width between ⅛ and 1 in. (3 and 25 mm); (*3*) a filled crack is not spalled or only lightly spalled, but the filler is in unsatisfactory condition; or (*4*) the area between the corner break and the joints is lightly cracked (see Fig. X2.6 and Fig. X2.7). Lightly cracked means one low-severity crack dividing the corner into two pieces.

X2.3.2.3 *H*—One of the following conditions exists: (*1*) filled or nonfilled crack is severely spalled, causing definite FOD potential; (*2*) a nonfilled crack has a mean width greater than approximately 1 in. (25 mm), creating a tire damage potential; or (*3*) the area between the corner break and the joints is severely cracked (see Fig. X2.8).

X2.3.3 *How to Count:*

X2.3.3.1 A distress slab is recorded as one slab if it contains a single corner break, contains more than one break of a particular severity, or contains two or more breaks of different severities. For two or more breaks, the highest level of severity should be recorded. For example, a slab containing both light and medium-severity corner breaks should be counted as one slab with a medium corner break. Crack widths should be measured between vertical walls, not in spalled areas of the crack.

X2.3.3.2 If the corner break is faulted ⅛ in. (3 mm) or more, increase severity to the next higher level. If the corner is faulted more than ½ in. (13 mm), rate the corner break at high

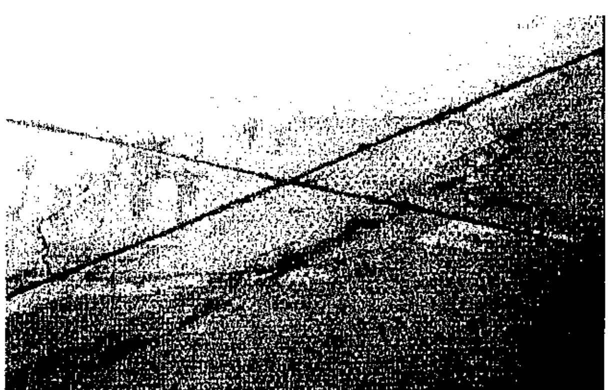


FIG. X2.4 Low-Severity Corner Break

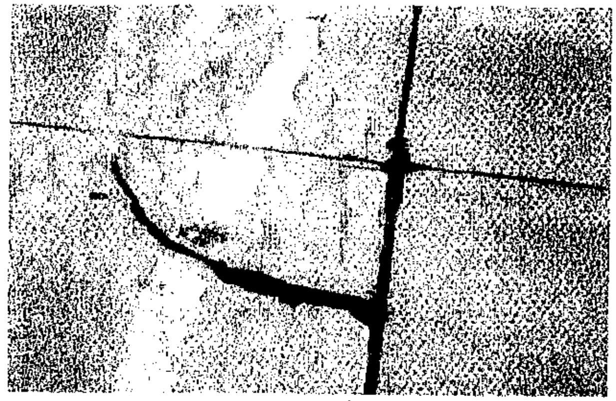


FIG. X2.5 Low-Severity Corner Break

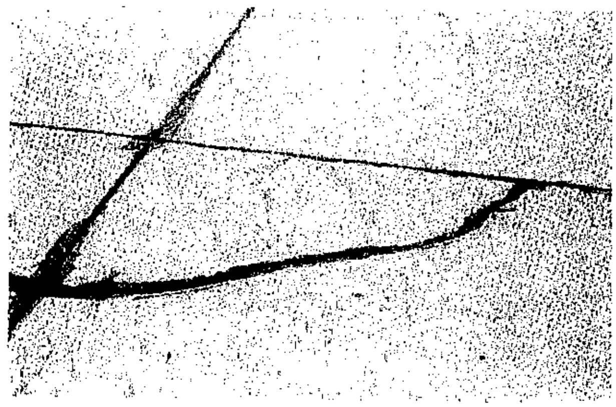


FIG. X2.6 Medium-Severity Corner Break (Area Between the Corner Break and the Joints Is Lightly Cracked)

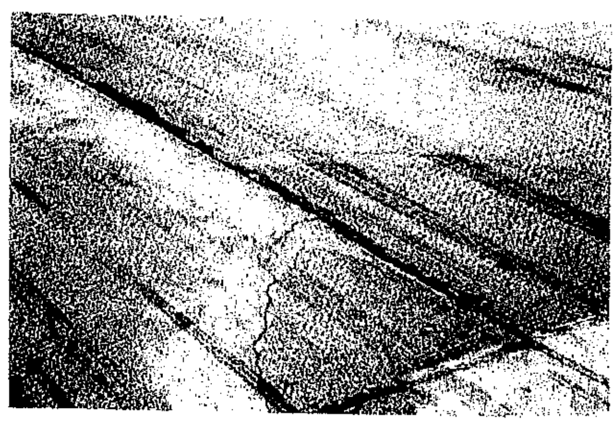


FIG. X2.7 Medium-Severity Corner Break

severity. If faulting in corner is incidental to faulting in the slab, rate faulting separately.

X2.3.3.3 The angle of crack into the slab is usually not evident at low severity. Unless the crack angle can be determined, to differentiate between the corner break and corner

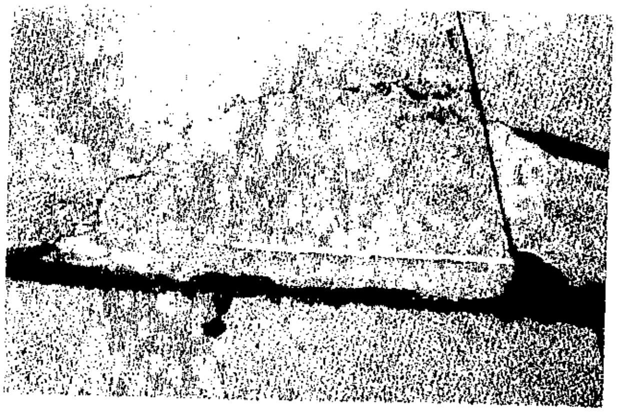


FIG. X2.8 High-Severity Corner Break

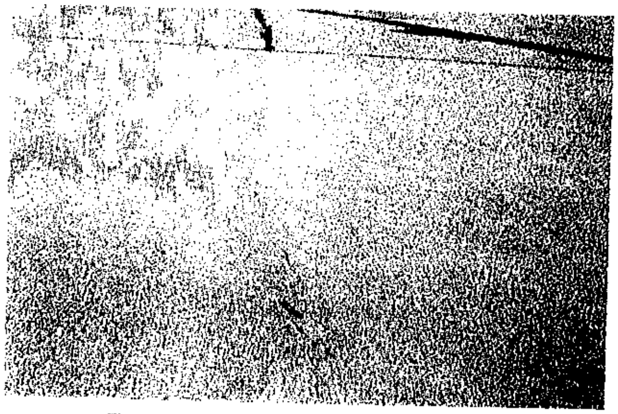


FIG. X2.9 Low-Severity Longitudinal Crack

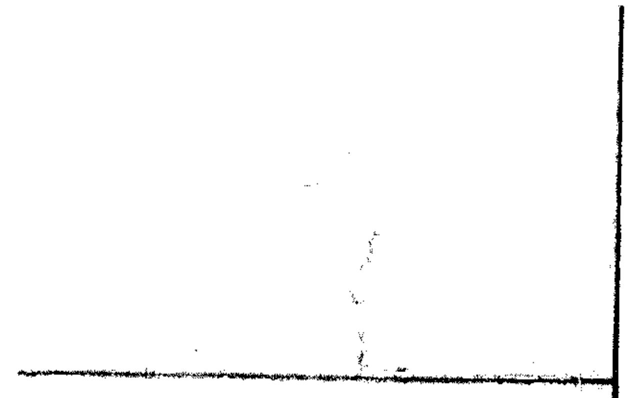


FIG. X2.10 Low-Severity Filled Longitudinal Cracks

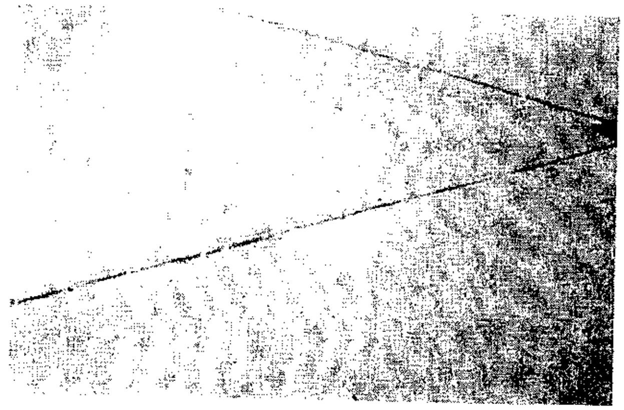


FIG. X2.11 Low-Severity Diagonal Crack

spall, use the following criteria. If the crack intersects both joints more than 2 ft (600 mm) from the corner, it is a corner break. If it is less than 2 ft, unless you can verify the crack is vertical, call it a spall.

X2.4 *Longitudinal, Transverse, and Diagonal Cracks:*

X2.4.1 *Description*—These cracks, that divide the slab into two or three pieces, are usually caused by a combination of load repetition, curling stresses, and shrinkage stresses. (For slabs divided into four or more pieces, see X2.13.) Low-severity cracks are usually warping- or friction-related and are not considered major structural distresses. Medium- or high-severity cracks are usually working cracks and are considered major structural distresses.

NOTE X2.3—Hairline cracks that are only a few feet long and do not extend across the entire slab are rated as shrinkage cracks.

X2.4.2 *Severity Levels:*

X2.4.2.1 *L*—Crack has little or minor spalling (no FOD potential). If nonfilled, it has a mean width less than approximately ⅛ in. (3 mm). A filled crack can be of any width, but the filler material must be in satisfactory condition; or the slab is divided into three pieces by low-severity cracks (see Figs. X2.9-X2.11).

X2.4.2.2 *M*—One of the following conditions exists: (*1*) filled or nonfilled crack is moderately spalled (some FOD potential); (*2*) a nonfilled crack has a mean width between ⅛ and 1 in. (3 and 25 mm); (*3*) a filled crack is not spalled or only lightly spalled, but the filler is in unsatisfactory condition; or (*4*) the slab is divided into three pieces by two or more cracks, one of which is at least medium severity (see Figs. X2.12-X2.14).

X2.4.2.3 *H*—One of the following conditions exists: (*1*) filled or nonfilled crack is severely spalled, causing definite FOD potential; (*2*) a nonfilled crack has a mean width greater than approximately 1 in. (25 mm), creating a tire damage potential; or (*3*) the slab is divided into three pieces by two or more cracks, one of which is at least high severity (see Figs. X2.15-X2.17).

X2.4.3 *How to Count:*

X2.4.3.1 Once the severity has been identified, the distress is recorded as one slab. If the slab is divided into four or more pieces by cracks, refer to the distress type given in X2.13.

X2.4.3.2 Cracks used to define and rate corner breaks, "D" cracks, patches, shrinkage cracks, and spalls are not recorded as L/T/D cracks.

X2.5 *Durability ("D") Cracking:*

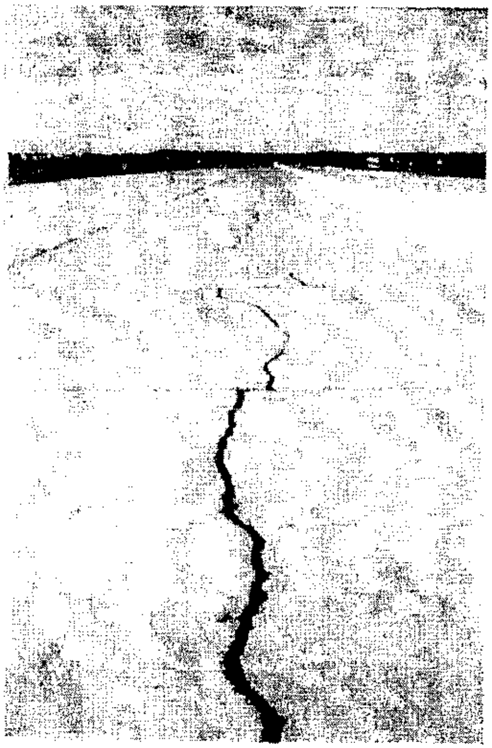


FIG. X2.12 Medium-Severity Longitudinal Crack

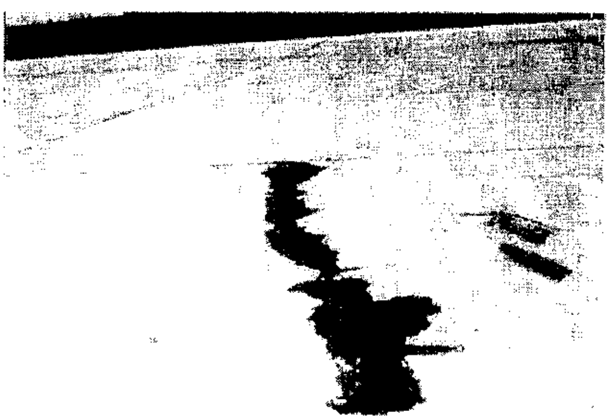


FIG. X2.13 Medium-Severity Transverse Crack

FIG. X2.14 Medium-Severity Transverse Crack

FIG. X2.15 High-Severity Crack

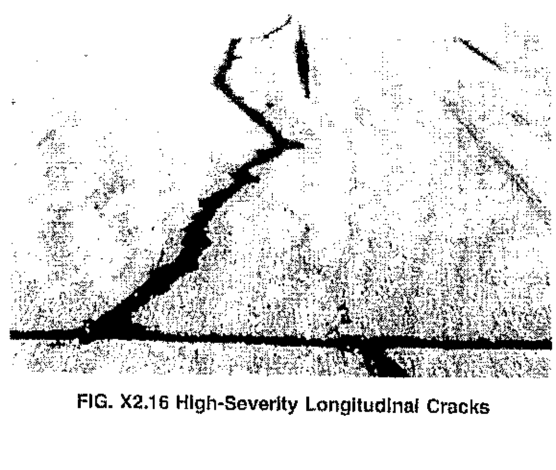


FIG. X2.16 High-Severity Longitudinal Cracks

X2.5.1 *Description*—Durability cracking is caused by the concrete's inability to withstand environmental factors, such as freeze-thaw cycles. It usually appears as a pattern of cracks running parallel to a joint or linear crack. A dark coloring can usually be seen around the fine durability cracks. This type of cracking may eventually lead to disintegration of the concrete within 1 to 2 ft (0.3 to 0.6 m) of the joint or crack.

X2.5.2 *Severity Levels*:

X2.5.2.1 *L*—"D" cracking is defined by hairline cracks occurring in a limited area of the slab, such as one or two corners or along one joint. Little or no disintegration has occurred. No FOD potential (see Fig. X2.18 and Fig. X2.19).

X2.5.2.2 *M*—"D" cracking has developed over a considerable amount of slab area with little or no disintegration or FOD potential; or "D" cracking has occurred in a limited area of the

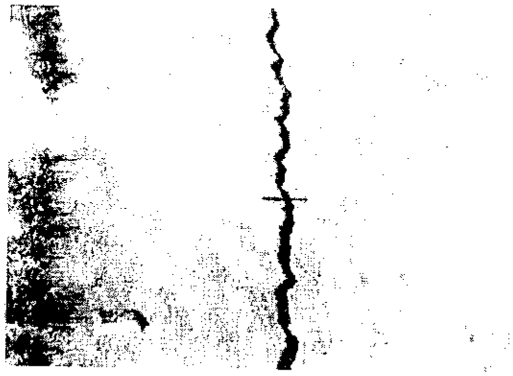


FIG. X2.17 High-Severity Crack

FIG. X2.18 Low-Severity "D" Cracking

NOTE 1—Slab is beginning to break up near corner.

FIG. X2.19 Low-Severity "D" Cracking Approaching Medium Severity

slab, such as one or two corners or along one joint, but pieces are missing and disintegration has occurred. Some FOD potential (see Fig. X2.20 and Fig. X2.21).

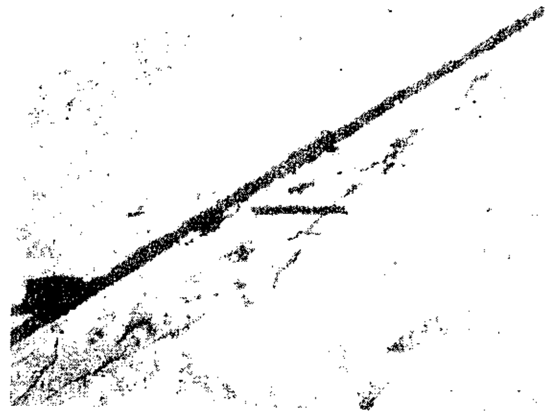


FIG. X2.20 Medium-Severity "D" Cracking

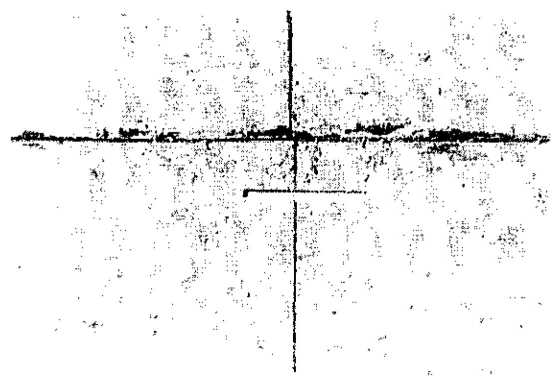


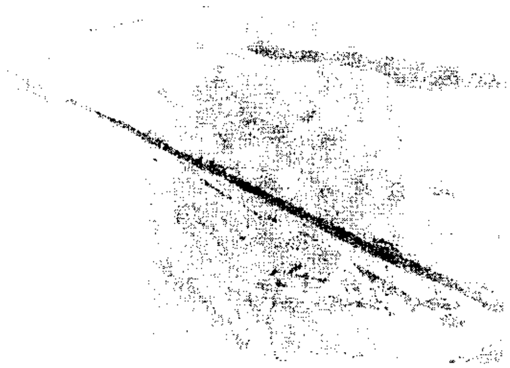
FIG. X2.21 Medium-Severity "D" Cracking Occurring in Limited Area of Slab

X2.5.2.3 *H*—"D" cracking has developed over a considerable amount of slab area with disintegration or FOD potential (see Fig. X2.22 and Fig. X2.23).

X2.5.3 *How to Count*—When the distress is located and rated at one severity, it is counted as one slab. If more than one severity level is found, the slab is counted as having the higher severity distress. For example, if low- and medium-durability cracking are located on one slab, the slab is counted as having medium only. If "D" cracking is counted, scaling on the same slab should not be recorded.

X2.6 *Joint Seal Damage:*

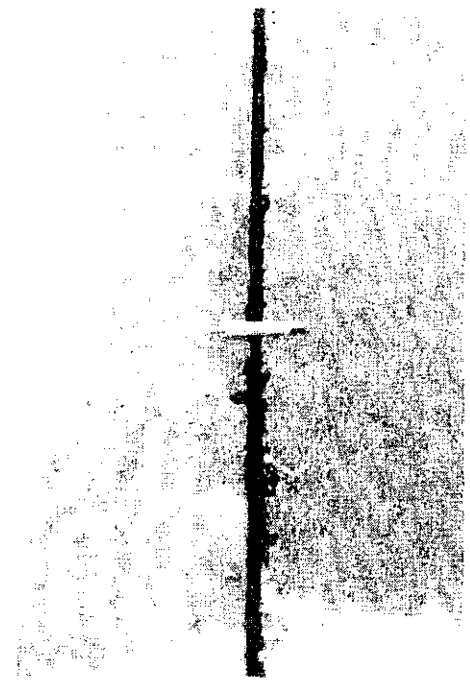
X2.6.1 *Description*—Joint seal damage is any condition that enables soil or rocks to accumulate in the joints or allows significant infiltration of water. Accumulation of incompressible materials prevents the slabs from expanding and may result in buckling, shattering, or spalling. A pliable joint filler bonded to the edges of the slabs protects the joints from accumulation of materials and also prevents water from seeping down and softening the foundation supporting the slab. Typical types of joint seal damage are: (*1*) stripping of joint



NOTE 1—The "D" cracking occurs over more than one joint with some disintegration.

FIG. X2.22 High-Severity "D" Cracking

FIG. X2.23 High-Severity "D" Cracking



NOTE 1—This condition existed on only a few joints in the pavement section. If all joint sealant were as shown, it would have been rated medium.

FIG. X2.24 Low-Severity Joint Seal Damage

sealant, (*2*) extrusion of joint sealant, (*3*) weed growth, (*4*) hardening of the filler (oxidation), (*5*) loss of bond to the slab edges, and (*6*) lack or absence of sealant in the joint.

X2.6.2 *Severity Levels*:

X2.6.2.1 *L*—Joint sealer is in generally good condition throughout the sample. Sealant is performing well with only a minor amount of any of the above types of damage present (see Fig. X2.24). Joint seal damage is at low severity if a few of the joints have sealer which has debonded from, but is still in contact with, the joint edge. This condition exists if a knife blade can be inserted between sealer and joint face without resistance.

X2.6.2.2 *M*—Joint sealer is in generally fair condition over the entire surveyed sample with one or more of the above types of damage occurring to a moderate degree. Sealant needs replacement within two years (see Fig. X2.25). Joint seal damage is at medium severity if a few of the joints have any of the following conditions: (*1*) joint sealer is in place, but water access is possible through visible openings no more than ⅛ in. (3 mm) wide. If a knife blade cannot be inserted easily between sealer and joint face, this condition does not exist; (2) pumping debris are evident at the joint; (*3*) joint sealer is oxidized and "lifeless" but pliable (like a rope), and generally fills the joint opening; or (*4*) vegetation in the joint is obvious, but does not obscure the joint opening.

X2.6.2.3 *H*—Joint sealer is in generally poor condition over the entire surveyed sample with one or more of the above types of damage occurring to a severe degree. Sealant needs immediate replacement (see Fig. X2.26 and Fig. X2.27). Joint seal damage is at high severity if 10 % or more of the joint sealer exceeds limiting criteria listed above, or if 10 % or more of sealer is missing.

X2.6.3 *How to Count*:

X2.6.3.1 Joint seal damage is not counted on a slab-by-slab basis, but is rated based on the overall condition of the sealant in the sample unit.

X2.6.3.2 Joint sealer is in satisfactory condition if it prevents entry of water into the joint, it has some elasticity, and if there is no vegetation growing between the sealer and joint face.

X2.6.3.3 Premolded sealer is rated using the same criteria as above except as follows: (*1*) premolded sealer must be elastic and must be firmly pressed against the joint walls; and (2) premolded sealer must be below the joint edge. If it extends above the surface, it can be caught by moving equipment such as snow plows or brooms and be pulled out of the joint. Premolded sealer is recorded at low severity if any part is visible above joint edge. It is at medium severity if 10 % or

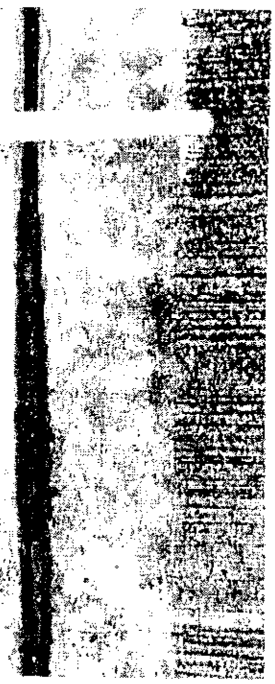


FIG. X2.25 Medium-Severity Joint Seal Damage (Note that Sealant has Lost Bond and Is Highly Oxidized)

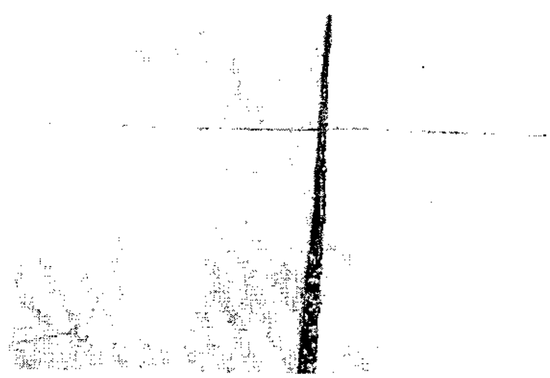


FIG. X2.26 High-Severity Joint Seal Damage (Complete Loss of Sealant; Joint Is Filled with Incompressible Material)

more of the length is above joint edge or if any part is more than ½ in. (12 mm) above joint edge. It is at high severity if 20 % or more is above joint edge or if any part is more than 1 in. (25 mm) above joint edge, or if 10 % or more is missing.

X2.6.3.4 Rate joint sealer by joint segment. Sample unit rating is the same as the most severe rating held by at least 20 % of segments rated.

X2.6.3.5 Rate only the left and upstation joints along sample unit boundaries.

X2.6.3.6 In rating oxidation, do not rate on appearance. Rate on resilience. Some joint sealer will have a very dull surface, and may even show surface cracks in the oxidized

FIG. X2.27 High-Severity Joint Seal Damage (Extensive Amount of Weed Growth)

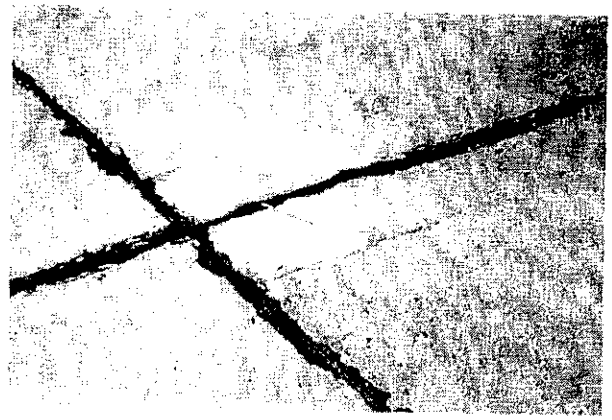


FIG. X2.28 Low-Severity Small Patch

layer. If the sealer is performing satisfactorily and has good characteristics beneath the surface, it is satisfactory.

X2.7 *Patching, Small (Less Than 5 ft^2(0.5 m^2)):*

X2.7.1 *Description*—A patch is an area where the original pavement has been removed and replaced by a filler material. For condition evaluation, patching is divided into two types: small (less than 5 ft^2 (0.5 m^2)) and large (over 5 ft^2). Large patches are described in the next section.

X2.7.2 *Severity Levels*:

X2.7.2.1 *L*—Patch is functioning well with little or no deterioration (see Fig. X2.28 and Fig. X2.29).

X2.7.2.2 *M*—Patch that has deterioration or moderate spalling, or both, can be seen around the edges. Patch material can be dislodged with considerable effort (minor FOD potential) (see Fig. X2.30 and Fig. X2.31).

X2.7.2.3 *H*—Patch deterioration, either by spalling around the patch or cracking within the patch, to a state that warrants replacement (see Fig. X2.32).

X2.7.3 *How to Count*:

X2.7.3.1 If one or more small patches having the same severity level are located in a slab, it is counted as one slab

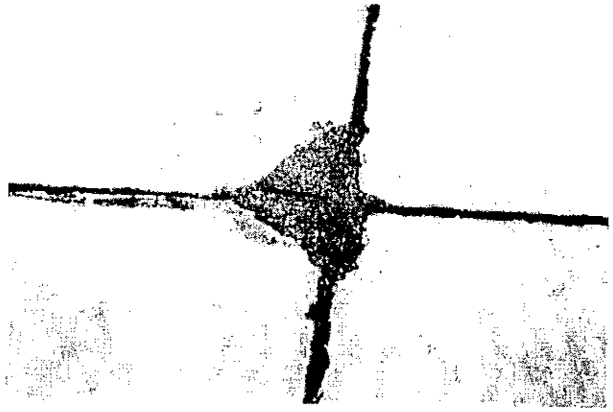


FIG. X2.29 Low-Severity Small Patch

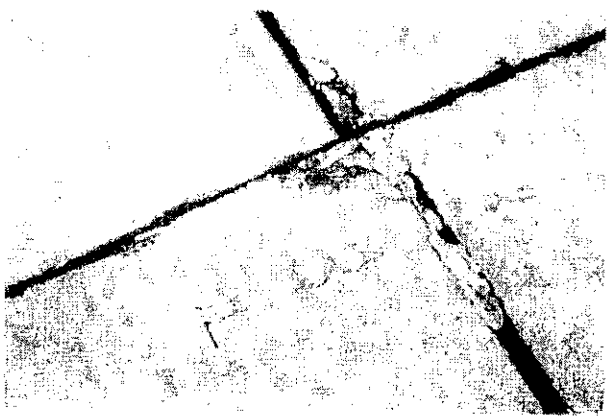


FIG. X2.30 Medium-Severity Small Patch

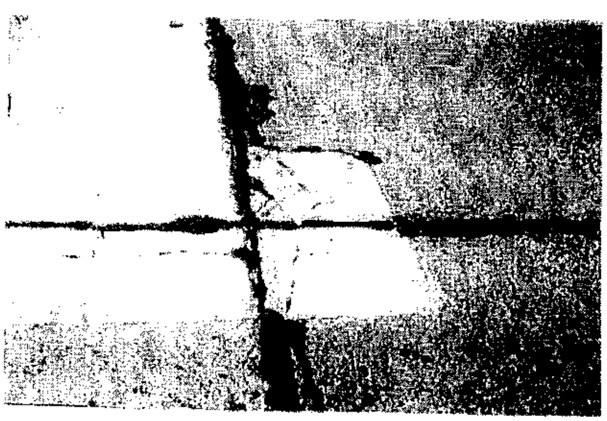


FIG. X2.31 Medium-Severity Small Patch

containing that distress. If more than one severity level occurs, it is counted as one slab with the higher severity level being recorded.

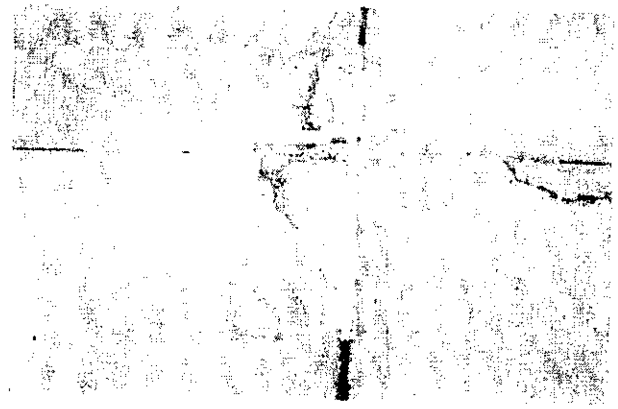


FIG. X2.32 High-Severity Small Patch

X2.7.3.2 If a crack is repaired by a narrow patch (that is, 4 to 10 in. (102 to 254 mm) wide), only the crack and not the patch should be recorded at the appropriate severity level.

X2.8 *Patching, Large (Over 5 ft² (0.5 m²)) and Utility Cut:*

X2.8.1 *Description*—Patching is the same as defined in the previous section. A utility cut is a patch that has replaced the original pavement because of placement of underground utilities. The severity levels of a utility cut are the same as those for regular patching.

X2.8.2 *Severity Levels:*

X2.8.2.1 *L*—Patch is functioning well with very little or no deterioration (see Figs. X2.33-X2.35).

X2.8.2.2 *M*—Patch deterioration or moderate spalling, or both, can be seen around the edges. Patch material can be dislodged with considerable effort, causing some FOD potential (see Fig. X2.36).

X2.8.2.3 *H*—Patch has deteriorated to a state that causes considerable roughness or high FOD potential, or both. The extent of the deterioration warrants replacement of the patch (see Fig. X2.37).

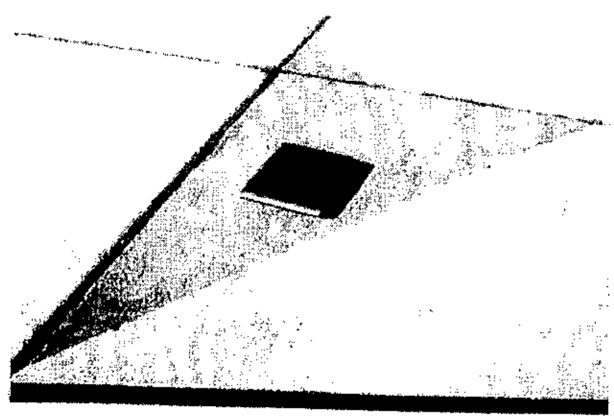


FIG. X2.33 Low-Severity Patch

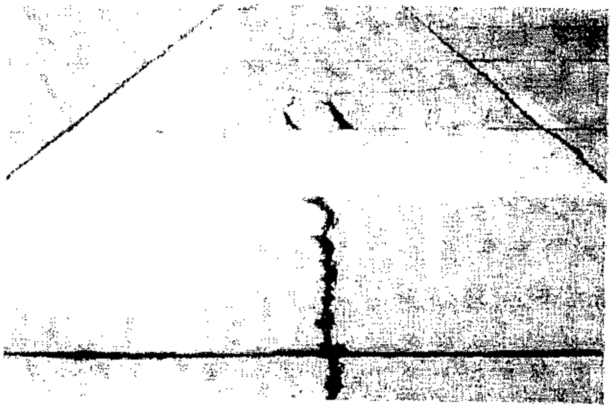


FIG. X2.34 Low-Severity Patch

FIG. X2.37 High-Severity Patch

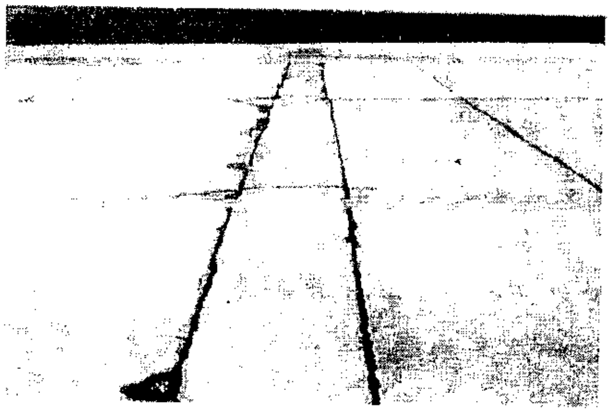


FIG. X2.35 Low-Severity Utility Cut

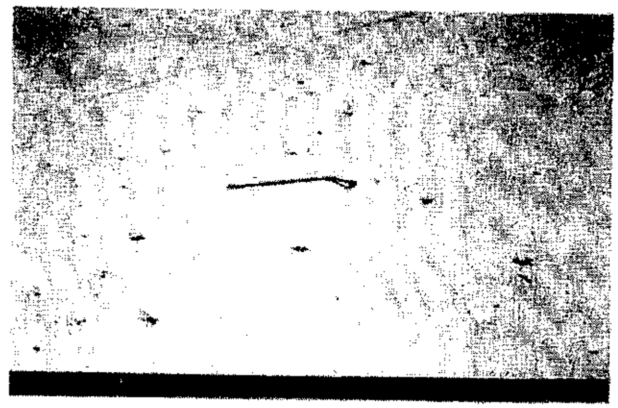


FIG. X2.38 Popouts

FIG. X2.36 Medium-Severity Utility Cut

X2.8.3 *How to Count*—The criteria are the same as for small patches.

X2.9 *Popouts:*

X2.9.1 *Description*—A popout is a small piece of pavement that breaks loose from the surface due to freeze-thaw action in combination with expansive aggregates. Popouts usually range from approximately 1 to 4 in. (25 to 100 mm) in diameter and from ½ to 2 in. (13 to 51 mm) deep.

X2.9.2 *Severity Levels*—No degrees of severity are defined for popouts. However, popouts must be extensive before they are counted as a distress; that is, average popout density must exceed approximately three popouts per square yard (per square metre) over the entire slab area (see Fig. X2.38).

X2.9.3 *How to Count*—The density of the distress must be measured. If there is any doubt about the average being greater than three popouts per square yard (per square metre), at least three random 1-yd^2 (1-m^2) areas should be checked. When the average is greater than this density, the slab is counted.

X2.10 *Pumping:*

X2.10.1 *Description*—Pumping is the ejection of material by water through joints or cracks caused by deflection of the slab under passing loads. As water is ejected, it carries particles of gravel, sand, clay, or silt resulting in a progressive loss of pavement support. Surface staining and base or subgrade material on the pavement close to joints or cracks are evidence of pumping. Pumping near joints indicates poor joint sealer and loss of support, which will lead to cracking under repeated loads. The joint seal must be identified as defective before pumping can be said to exist. Pumping can occur at cracks as well as joints.

X2.10.2 *Severity Levels*—No degrees of severity are defined. It is sufficient to indicate that pumping exists (see Figs. X2.39-X2.42).

X2.10.3 *How to Count*—Slabs are counted as follows: (see Fig. X2.43) one pumping joint between two slabs is counted as two slabs. However, if the remaining joints around the slab are also pumping, one slab is added per additional pumping joint.

X2.11 *Scaling, Map Cracking, and Crazing:*

X2.11.1 *Description*—Map cracking or crazing refers to a network of shallow, fine, or hairline cracks that extend only through the upper surface of the concrete. The cracks tend to intersect at angles of 120°. Map cracking or crazing is usually caused by over finishing the concrete and may lead to scaling of the surface, that is the breakdown of the slab surface to a depth of approximately ¼ to ½ in. (6 to 13 mm). Scaling may also be caused by deicing salts, improper construction, freeze-thaw cycles, and poor aggregate. Another recognized source of distress is the reaction between the alkalies (Na_2O and K_2O) in some cements and certain minerals in some aggregates. Products formed by the reaction between the alkalies and aggregate result in expansions that cause a breakdown in the concrete. This generally occurs throughout the slab and not just at joints where "D" cracking normally occurs.

X2.11.2 *Severity Levels*:

X2.11.2.1 *L*—Crazing or map cracking exists over significant slab area. The surface is in good condition with no scaling. The crack pattern must be well defined and easily recognized. Individual cracks should show some evidence of wear. Very early stages are ignored.

NOTE X2.4—The low-severity level is an indicator that scaling may develop in the future (see Fig. X2.44).

X2.11.2.2 *M*—Slab is scaled over approximately 5 % or less of the surface with some FOD potential (see Fig. X2.45).

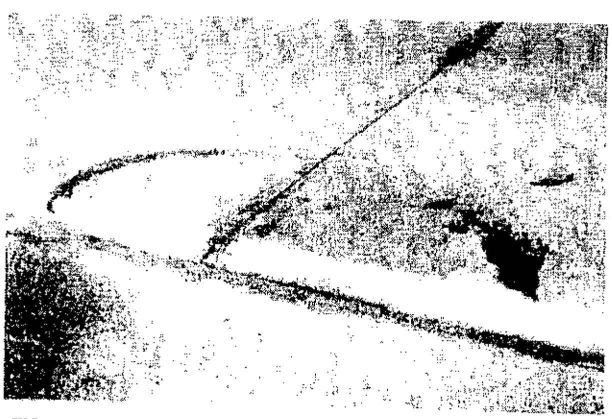


FIG. X2.39 Pumping (Note Fine Material on Surface That has Been Pumped Out Causing Corner Break)

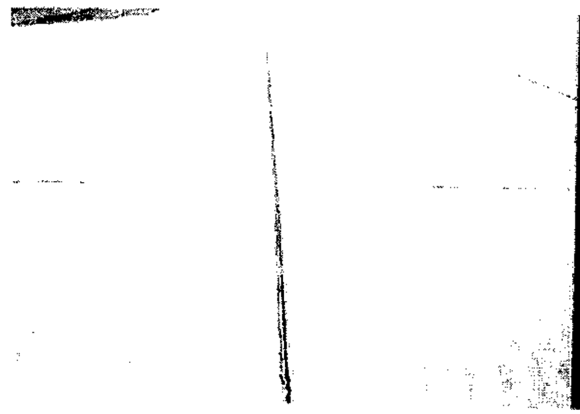


FIG. X2.40 Pumping (Note Stains on Pavement)

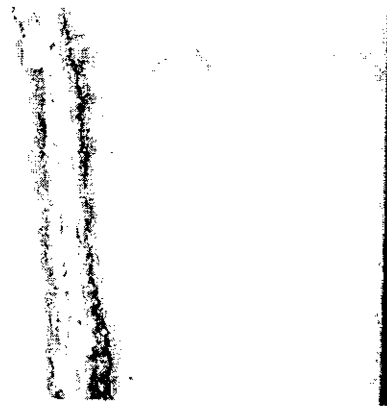


FIG. X2.41 Pumping (Close-Up of Fine Materials Collecting in the Joint)

FIG. X2.42 Pumping

X2.11.2.3 *H*—Slab is severely scaled causing a high FOD potential. Usually, more than 5 % of the surface is affected (see Figs. X2.46-X2.48).

X2.11.3 *How to Count*—If two or more levels of severity exist on a slab, the slab is counted as one slab having the

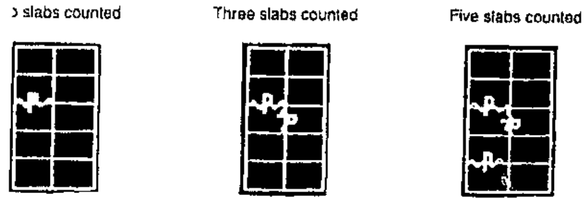


FIG. X2.43 Slab Counting Procedure for Distresses

FIG. X2.44 Low-Severity Crazing

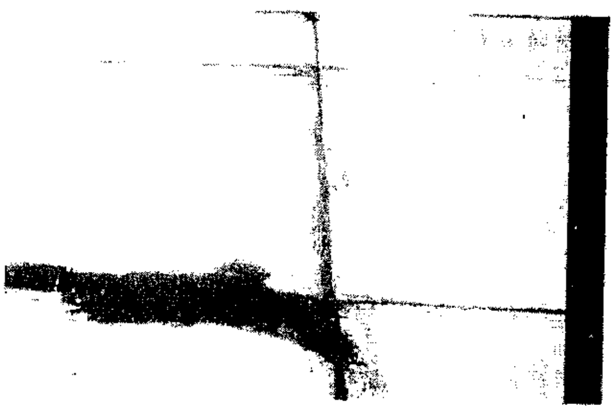


FIG. X2.45 Medium-Severity Scaling

maximum level of severity. For example, if both low-severity crazing and medium scaling exist on one slab, the slab is counted as one slab containing medium scaling. If "D" cracking is counted, scaling is not counted.

X2.12 *Settlement or Faulting:*

X2.12.1 *Description*—Settlement or faulting is a difference of elevation at a joint or crack caused by upheaval or consolidation.

X2.12.2 *Severity Levels*—Severity levels are defined by the difference in elevation across the fault and the associated decrease in ride quality and safety as severity increases:

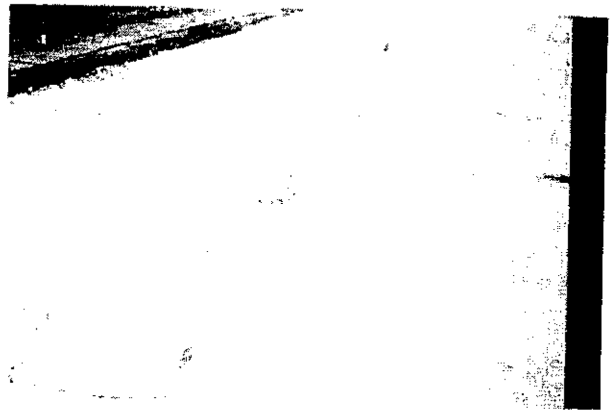


FIG. X2.46 High-Severity Scaling

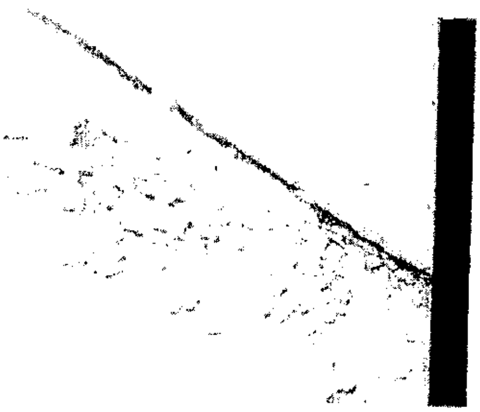


FIG. X2.47 Close-Up of High-Severity Scaling

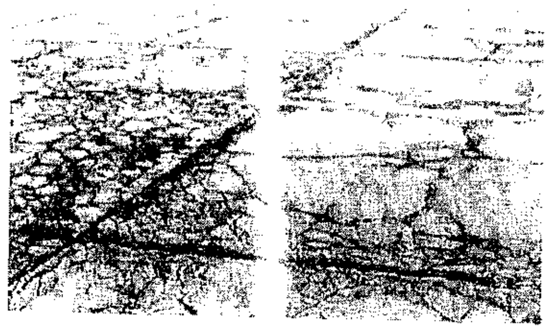


FIG. X2.48 High-Severity Scaling Caused by Alkali Aggregate Reaction

	Difference in Elevation		
	Runways/Taxiways	Aprons	Figures
L	< ¼ in. (6 mm)	⅛ < ½ in. (3 to 13 mm)	Fig. X2.49 and Fig. X2.50
M	¼ to ½ in. (6 to 13 mm)	½ to 1 in. (13 to 25 mm)	Fig. X2.51
H	> ½ in. (13 mm)	> 1 in. (25 mm)	Fig. X2.52 and Fig. X2.53

X2.12.3 *How to Count:*

X2.12.3.1 In counting settlement, a fault between two slabs is counted as one slab. A straightedge or level should be used to aid in measuring the difference in elevation between the two slabs.

X2.12.3.2 Construction-induced elevation differential is not rated in PCI procedures. Where construction differential exists, it can often be identified by the way the high side of the joint was rolled down by finishers (usually within 6 in. (150 mm) of the joint) to meet the low-slab elevation.

X2.13 *Shattered Slab/Intersecting Cracks:*

X2.13.1 *Description*—Intersecting cracks are cracks that break the slab into four or more pieces due to overloading or inadequate support, or both. The high-severity level of this distress type, as defined as follows, is referred to as shattered slab. If all pieces or cracks are contained within a corner break, the distress is categorized as a severe corner break.

X2.13.2 *Severity Levels*:

X2.13.2.1 *L*—Slab is broken into four or five pieces predominantly defined by low-severity cracks (see Fig. X2.54 and Fig. X2.55).

X2.13.2.2 *M*—Slab is broken into four or five pieces with over 15 % of the cracks of medium severity (no high-severity cracks); slab is broken into six or more pieces with over 85 % of the cracks of low severity (see Fig. X2.56 and Fig. X2.57).

X2.13.2.3 *H*—At this level of severity, the slab is called shattered: (*1*) slab is broken into four or five pieces with some or all cracks of high severity; or (2) slab is broken into six or more pieces with over 15 % of the cracks of medium or high severity (see Fig. X2.58).

X2.13.3 *How to Count*—No other distress such as scaling, spalling, or durability cracking should be recorded if the slab is medium- or high-severity level since the severity of this distress would affect the slab's rating substantially. Shrinkage cracks should not be counted in determining whether or not the slab is broken into four or more pieces.

X2.14 *Shrinkage Cracks:*

X2.14.1 *Description*—Shrinkage cracks are hairline cracks that are usually only a few feet (centimetres) long and do not extend across the entire slab. They are formed during the setting and curing of the concrete and usually do not extend through the depth of the slab.

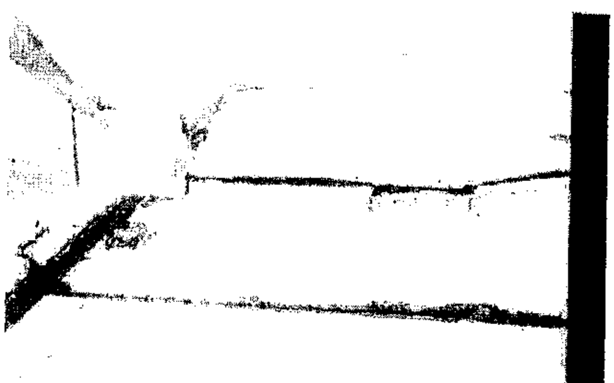


FIG. X2.49 Low-Severity Settlement (3/8 in. (9 mm)) on Apron

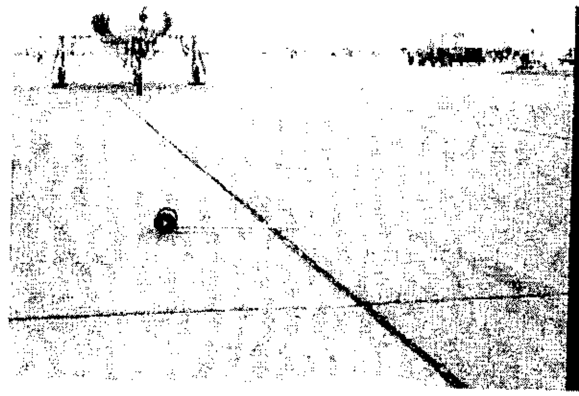


FIG. X2.50 Low-Severity Settlement on Apron

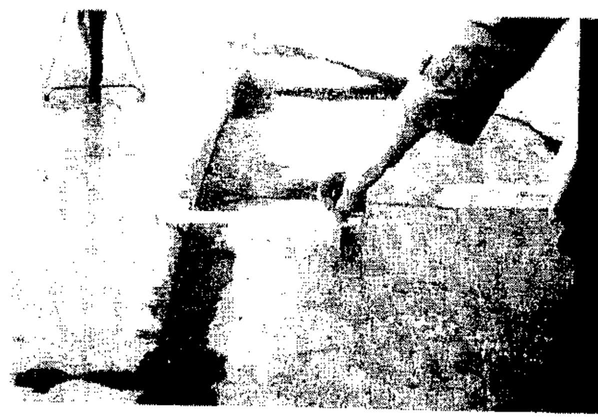


FIG. X2.51 Medium-Severity Settlement on Apron (>1/2 in. (13 mm))

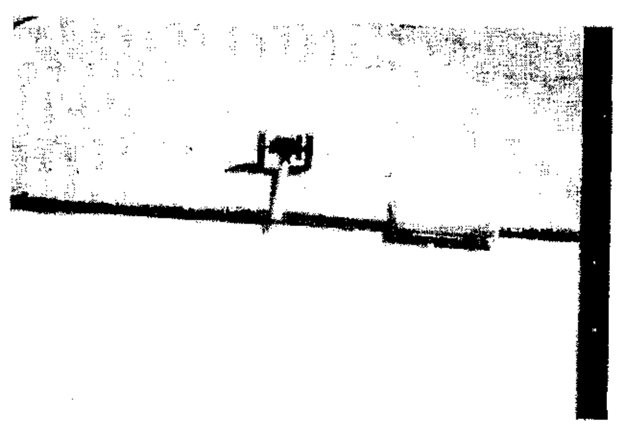


FIG. X2.52 High-Severity Settlement on Taxiway/Runway (3/4 in. (18 mm))

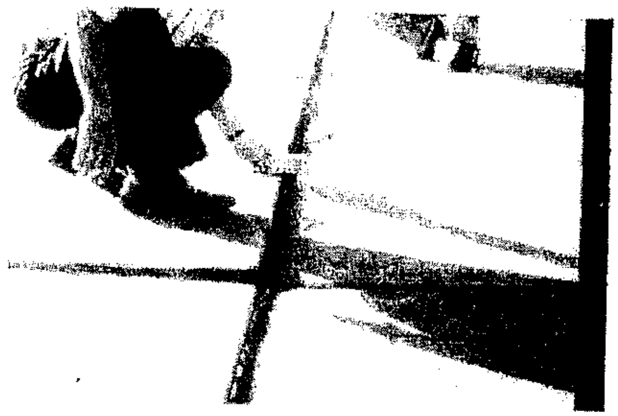


FIG. X2.53 High-Severity Settlement

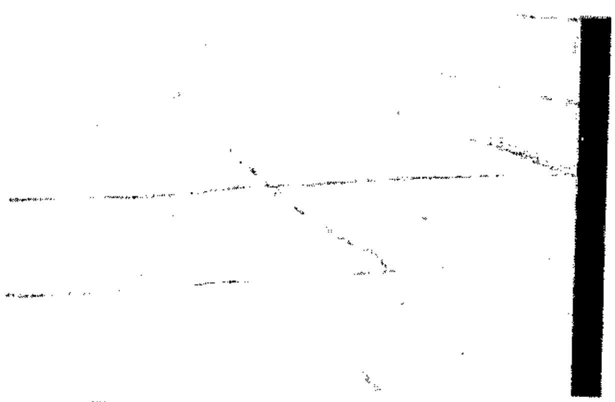


FIG. X2.54 Low-Severity Intersecting Cracks

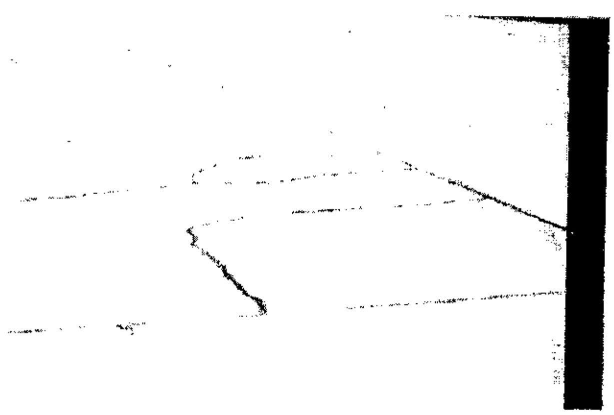


FIG. X2.55 Low-Severity Intersecting Cracks

X2.14.2 *Severity Levels*—No degrees of severity are defined. It is sufficient to indicate that shrinkage cracks exist (see Figs. X2.59-X2.61).

X2.14.3 *How to Count*—If one or more shrinkage cracks exist on one particular slab, the slab is counted as one slab with shrinkage cracks.

FIG. X2.56 Medium-Severity Intersecting Cracks

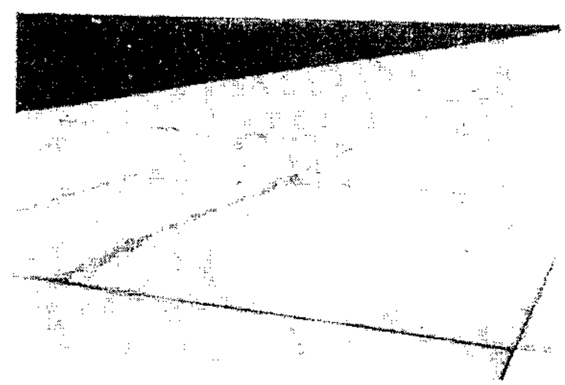


FIG. X2.57 Medium-Severity Intersecting Cracks

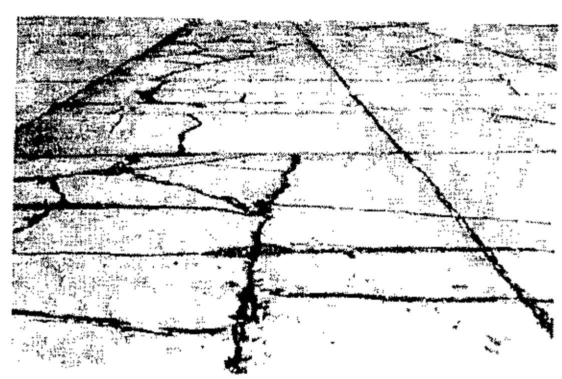


FIG. X2.58 Shattered Slab

X2.15 *Spalling (Transverse and Longitudinal Joint):*

X2.15.1 *Description*—Joint spalling is the breakdown of the slab edges within 2 ft (0.6 m) of the side of the joint. A joint spall usually does not extend vertically through the slab but intersects the joint at an angle. Spalling results from excessive stresses at the joint or crack caused by infiltration of incompressible materials or traffic load. Weak concrete at the joint (caused by overworking) combined with traffic loads is another cause of spalling.

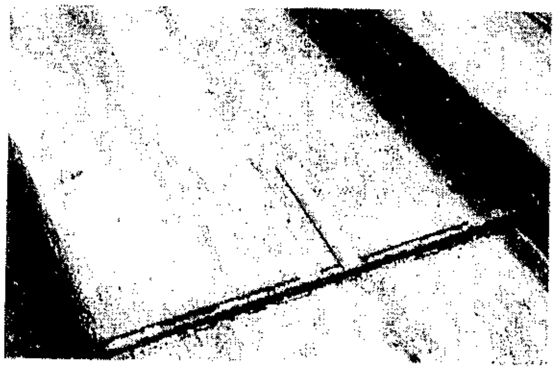


FIG. X2.59 Shrinkage Crack

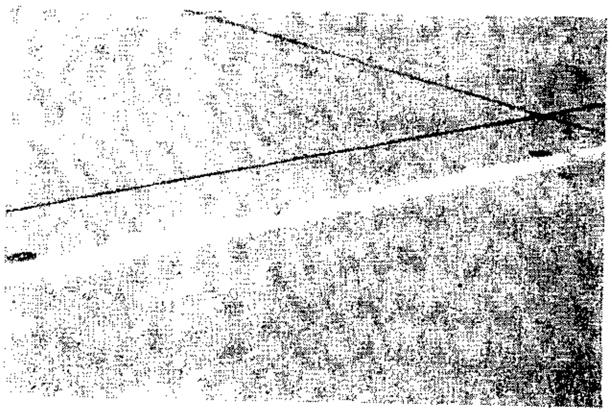


FIG. X2.60 Shrinkage Cracks

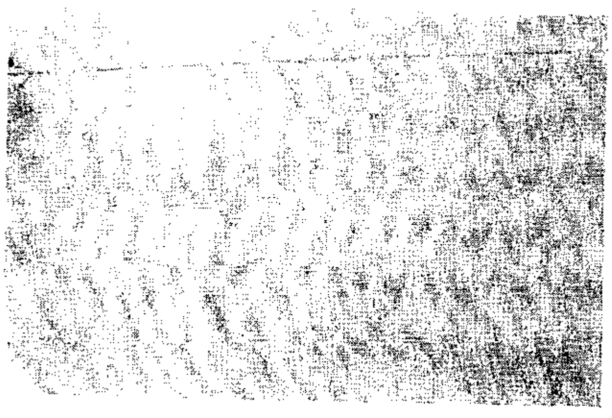


FIG. X2.61 Shrinkage Cracks

NOTE X2.5—Frayed condition as used in this test method indicates material is no longer in place along a joint or crack. Spalling indicates material may or may not be missing along a joint or crack.

X2.15.2 *Severity Levels*:

X2.15.2.1 *L*—Spall over 2 ft (0.6 m) long: (*1*) spall is broken into no more than three pieces defined by low- or medium-severity cracks; little or no FOD potential exists; or (*2*) joint is lightly frayed; little or no FOD potential (see Fig. X2.62 and Fig. X2.63). Spall less than 2 ft long is broken into pieces or fragmented with little FOD or tire damage potential exists (see Fig. X2.64).

X2.15.2.2 Lightly frayed means the upper edge of the joint is broken away leaving a spall no wider than 1 in. (25 mm) and no deeper than ½ in. (13 mm). The material is missing and the joint creates little or no FOD potential.

X2.15.2.3 *M*—Spall over 2 ft (0.6 m) long: (*1*) spall is broken into more than three pieces defined by light or medium cracks; (*2*) spall is broken into no more than three pieces with one or more of the cracks being severe with some FOD potential existing; or (*3*) joint is moderately frayed with some FOD potential (see Fig. X2.65). Spall less than 2 ft long: spall is broken into pieces or fragmented with some of the pieces loose or absent, causing considerable FOD or tire damage potential (see Fig. X2.66).

X2.15.2.4 Moderately frayed means the upper edge of the joint is broken away leaving a spall wider than 1 in. (25 mm) or deeper than ½ in. (13 mm). The material is mostly missing with some FOD potential.

X2.15.2.5 *H*—Spall over 2 ft (0.6 m) long: (*1*) spall is broken into more than three pieces defined by one or more high-severity cracks with high FOD potential and high possibility of the pieces becoming dislodged, or (*2*) joint is severely frayed with high FOD potential (see Fig. X2.67 and Fig. X2.68).

FIG. X2.62 Low-Severity Joint Spalling (If the Frayed Area was Less Than 2 ft (0.6 m) Long It Would not be Counted)

FIG. X2.63 Low-Severity Joint Spall

FIG. X2.64 Low-Severity Joint Spall

X2.15.3 *How to Count*—If the joint spall is located along the edge of one slab, it is counted as one slab with joint spalling. If spalling is located on more than one edge of the same slab, the edge having the highest severity is counted and recorded as one slab. Joint spalling can also occur along the edges of two adjacent slabs. If this is the case, each slab is counted as having joint spalling. If a joint spall is small enough, less than 3 in. (76 mm) wide, to be filled during a joint seal repair, it should not be recorded.

NOTE X2.6—If less than 2 ft (0.6 m) of the joint is lightly frayed, the spall should not be counted.

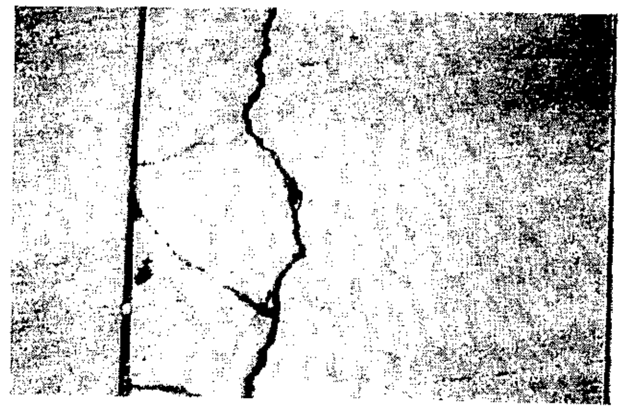


FIG. X2.65 Medium-Severity Joint Spall

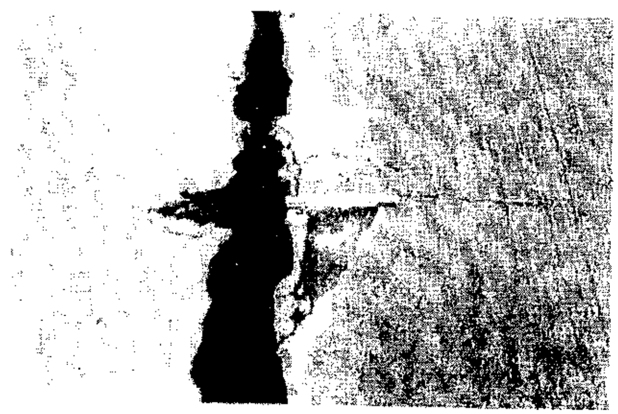


FIG. X2.66 Medium-Severity Joint Spall

FIG. X2.67 High-Severity Joint Spall

X2.16 *Spalling (Corner):*

X2.16.1 *Description*—Corner spalling is the raveling or breakdown of the slab within approximately 2 ft (0.6 m) of the

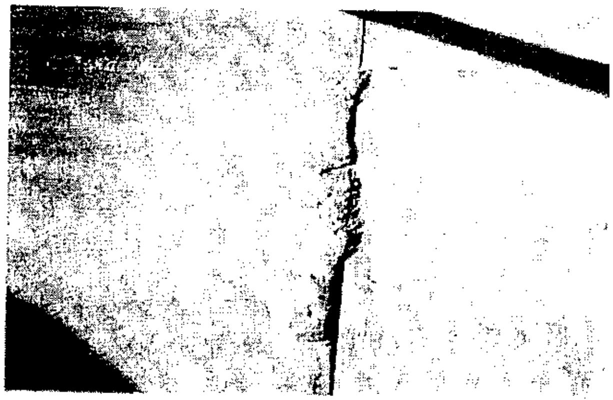


FIG. X2.68 High-Severity Joint Spall

FIG. X2.70 Low-Severity Corner Spall

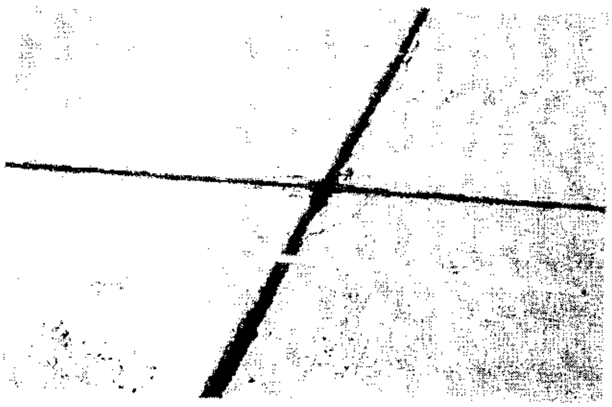


FIG. X2.69 Low-Severity Corner Spall

FIG. X2.71 Medium-Severity Corner Spall

corner. A corner spall differs from a corner break in that the spall usually angles downward to intersect the joint, while a break extends vertically through the slab.

X2.16.2 *Severity Levels*:

X2.16.2.1 *L*—One of the following conditions exists: (*1*) spall is broken into one or two pieces defined by low-severity cracks (little or no FOD potential); or (*2*) spall is defined by one medium-severity crack (little or no FOD potential) (see Fig. X2.69 and Fig. X2.70).

X2.16.2.2 *M*—One of the following conditions exists: (*1*) spall is broken into two or more pieces defined by medium-severity crack(s), and a few small fragments may be absent or loose; (*2*) spall is defined by one severe, fragmented crack that may be accompanied by a few hairline cracks; or, (*3*) spall has deteriorated to the point where loose material is causing some FOD potential (see Fig. X2.71 and Fig. X2.72).

X2.16.2.3 *H*—One of the following conditions exists: (*1*) spall is broken into two or more pieces defined by high-severity fragmented crack(s) with loose or absent fragments; (*2*) pieces of the spall have been displaced to the extent that a tire damage hazard exists; or (*3*) spall has deteriorated to the point where loose material is causing high FOD potential (see Fig. X2.73 and Fig. X2.74).

X2.16.3 *How to Count*:

X2.16.3.1 If one or more corner spalls having the same severity level are located in a slab, the slab is counted as one slab with corner spalling. If more than one severity level occurs, it is counted as one slab having the higher severity level.

X2.16.3.2 A corner spall smaller than 3 in. (76 mm) wide, measured from the edge of the slab, and filled with sealant is not recorded.

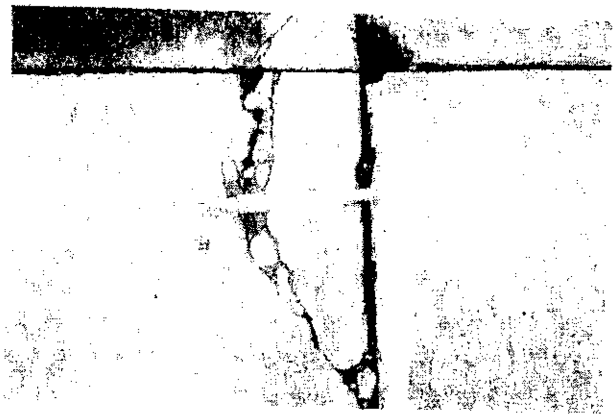


FIG. X2.72 Medium-Severity Corner Spall

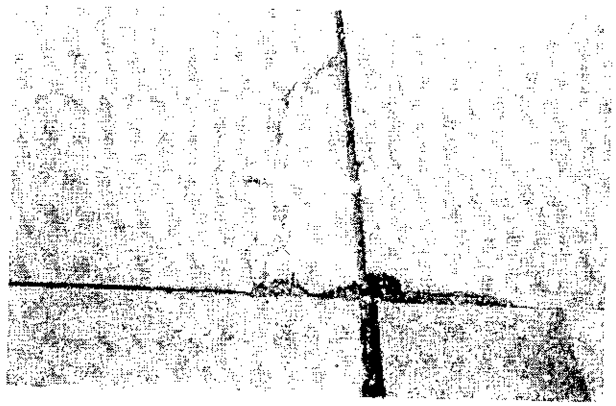


FIG. X2.73 High-Severity Corner Spall

FIG. X2.74 High-Severity Corner Spall

X3. AC PAVEMENT DEDUCT CURVES

X3.1 See Figs. X3.1-X3.19.

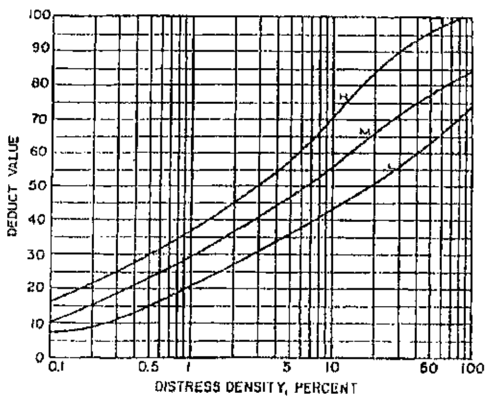


FIG. X3.1 Distress 1, Alligator Cracking

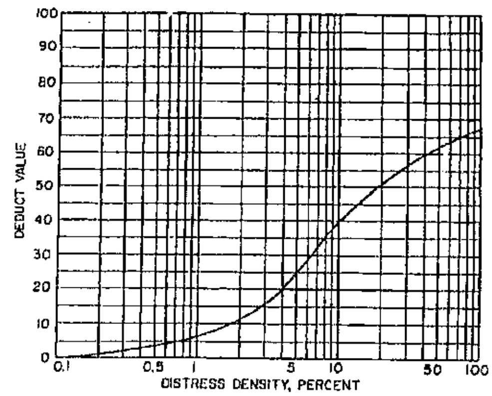


FIG. X3.2 Distress 2, Bleeding

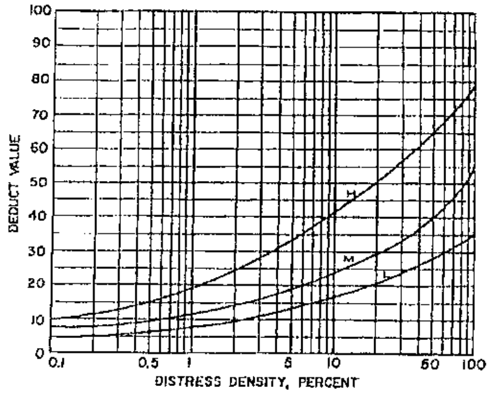


FIG. X3.3 Distress 3, Block Cracking

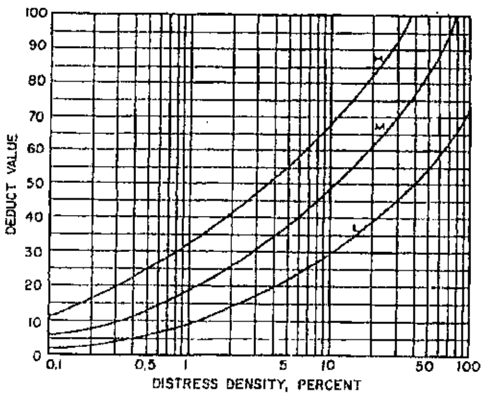


FIG. X3.4 Distress 4, Corrugation

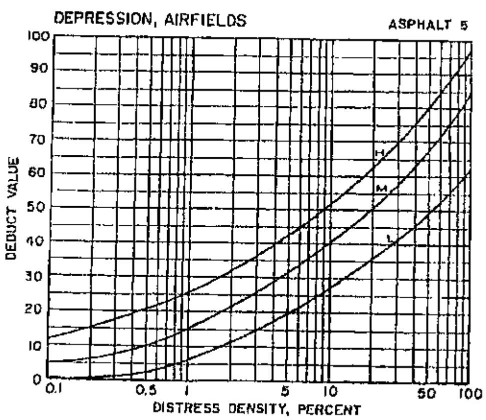


FIG. X3.5 Depression

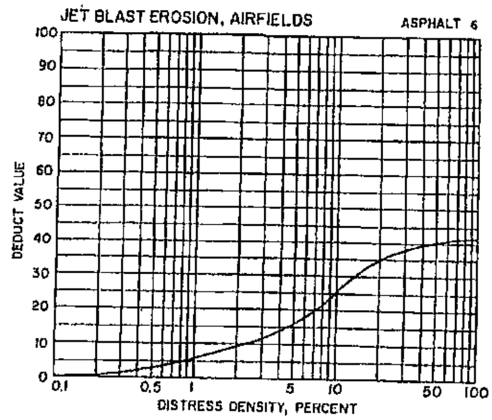


FIG. X3.6 Jet Blast

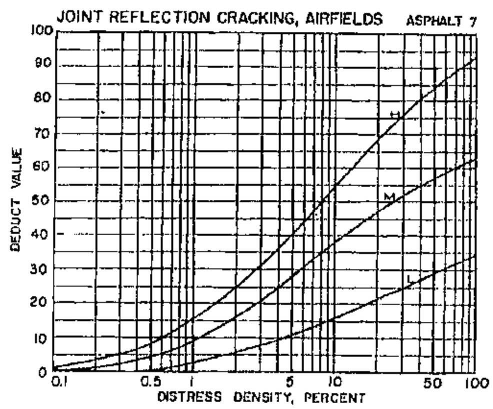


FIG. X3.7 Joint Reflection Cracking

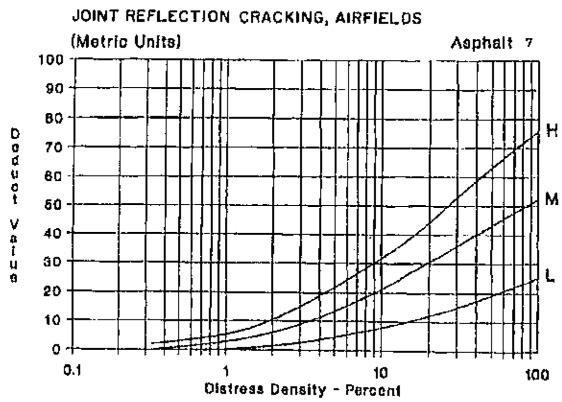


FIG. X3.8 Joint Reflection Cracking (Metric)

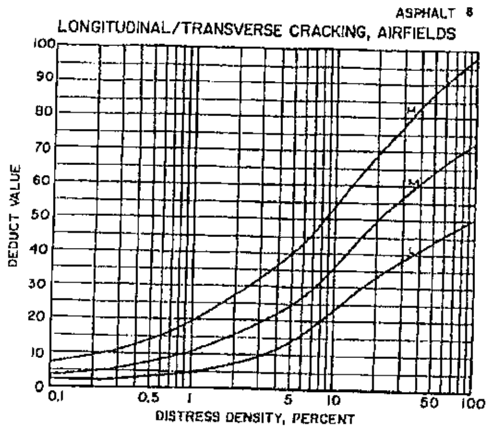


FIG. X3.9 Longitudinal/Transverse Cracking

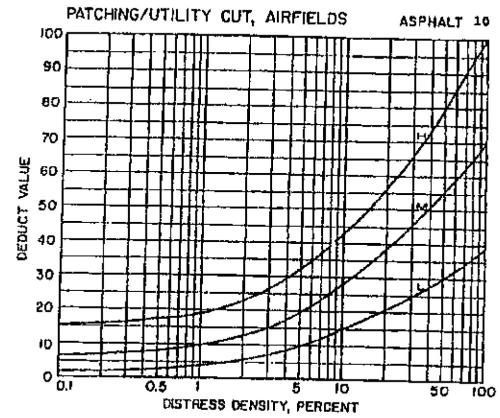


FIG. X3.12 Patching

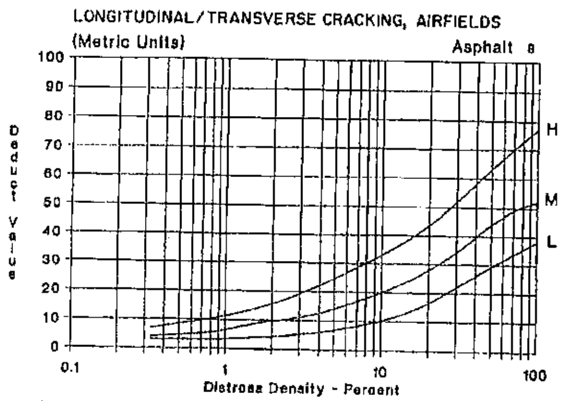


FIG. X3.10 Longitudinal/Transverse Cracking (Metric)

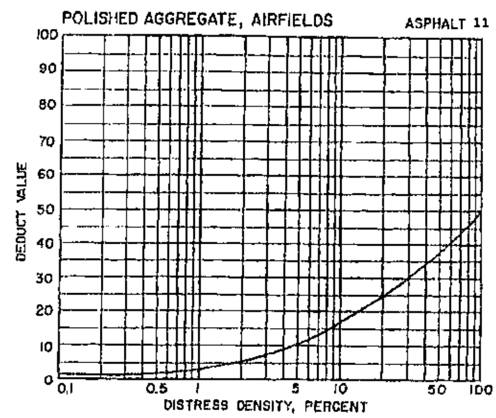


FIG. X3.13 Polished Aggregate

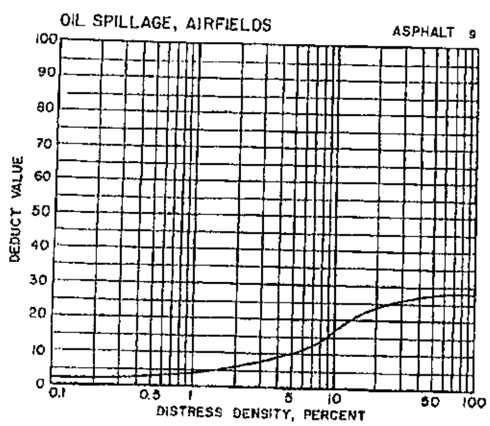


FIG. X3.11 Oil Spillage

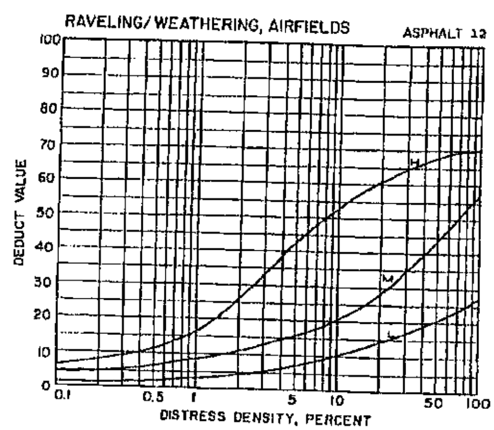


FIG. X3.14 Weathering/Raveling

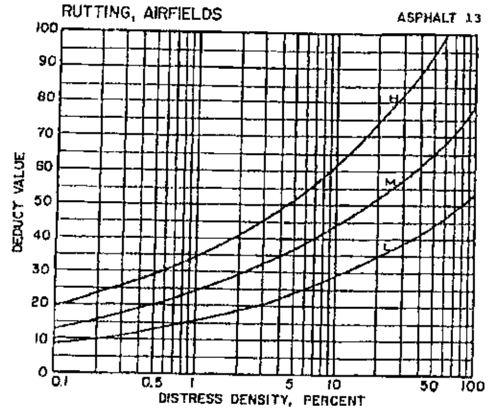


FIG. X3.15 Rutting

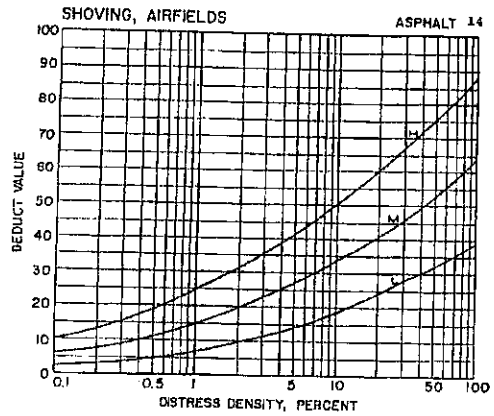


FIG. X3.16 Shoving

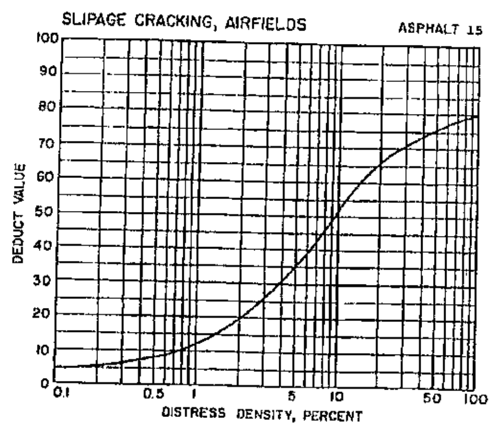


FIG. X3.17 Slippage Cracking

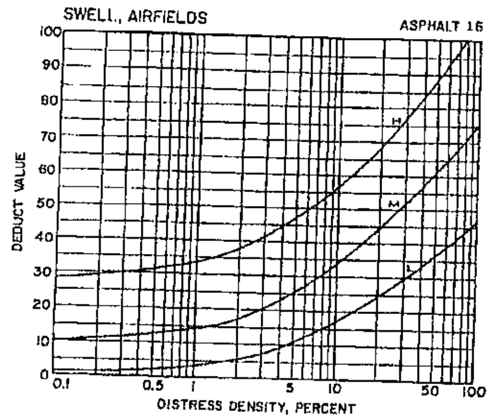


FIG. X3.18 Swelling

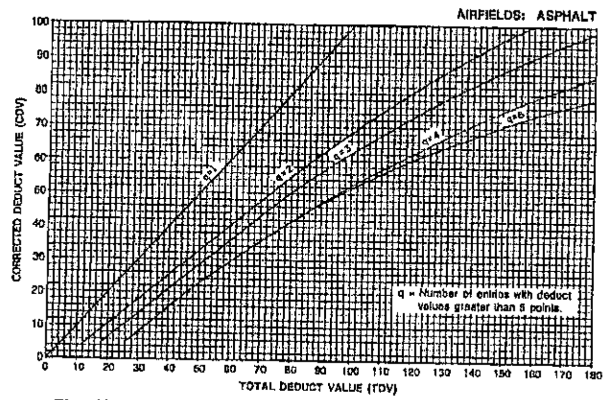


FIG. X3.19 Corrected DVs for Flexible Airfield Pavement

X4. PCC PAVEMENT DEDUCT CURVES

X4.1 See Figs. X4.1-X4.16.

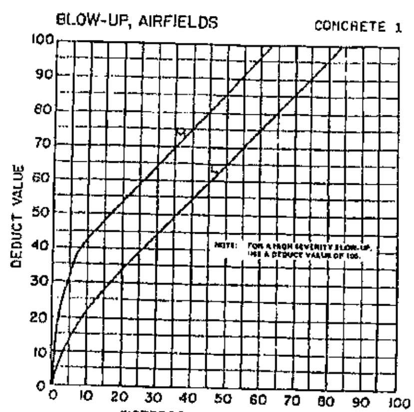


FIG. X4.1 Blowup

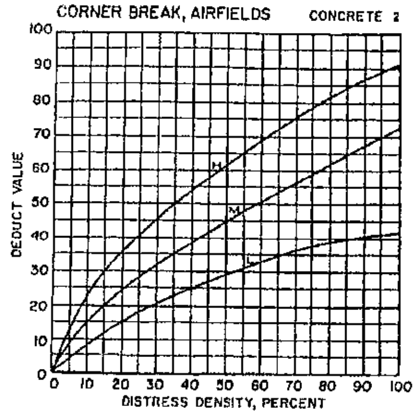


FIG. X4.2 Corner Break

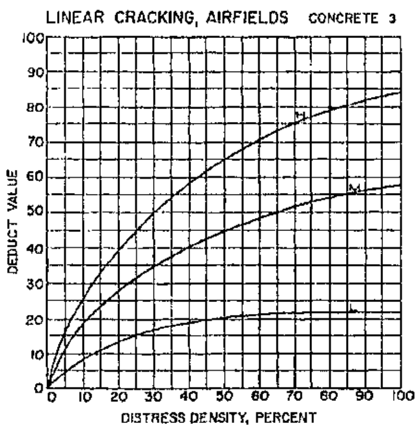


FIG. X4.3 Linear Cracking

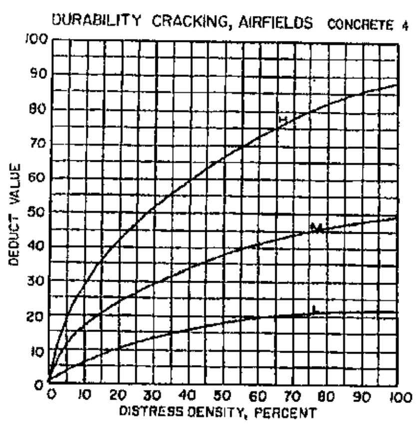


FIG. X4.4 Durability Cracking

JOINT SEAL DAMAGE **CONCRETE 5**

Joint seal damage is not rated by density. The severity of the distress is determined by the sealant's overall condition for a particular section.

The deduct values for the three levels of severity are as follows:

1. High Severity - 12 Points
2. Medium Severity - 7 Points
3. Low Severity - 2 Points

FIG. X4.5 Joint Seal Damage

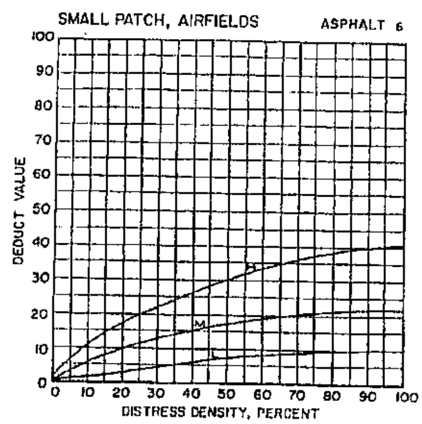


FIG. X4.6 Small Patch

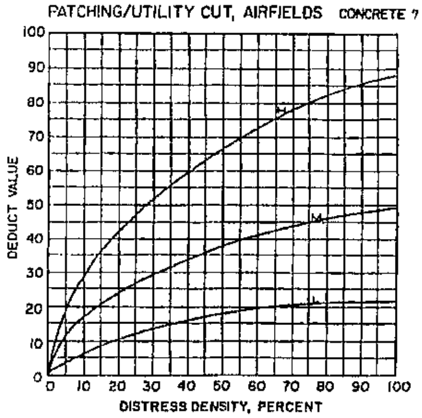


FIG. X4.7 Patching/Utility Cut

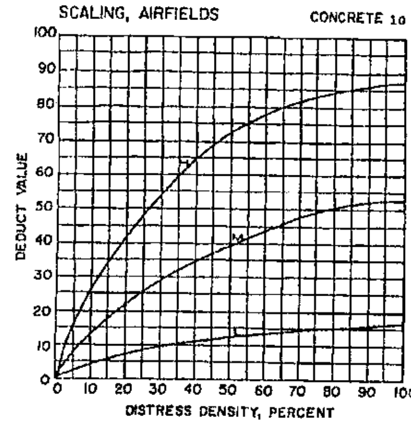


FIG. X4.10 Scaling/Crazing

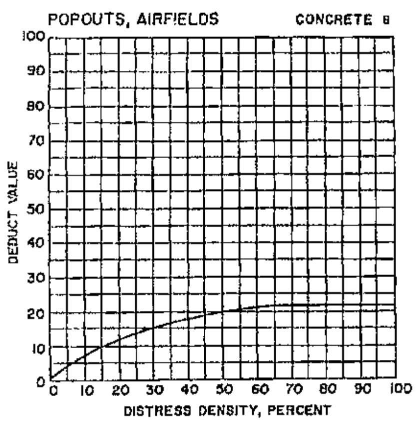


FIG. X4.8 Popouts

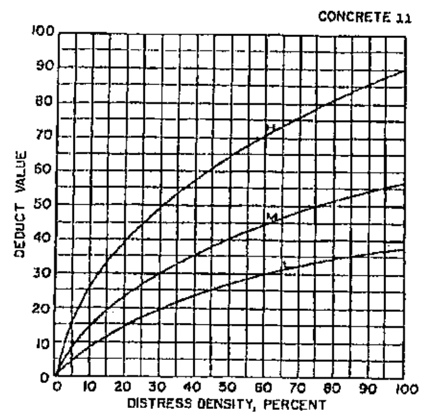


FIG. X4.11 Faulting

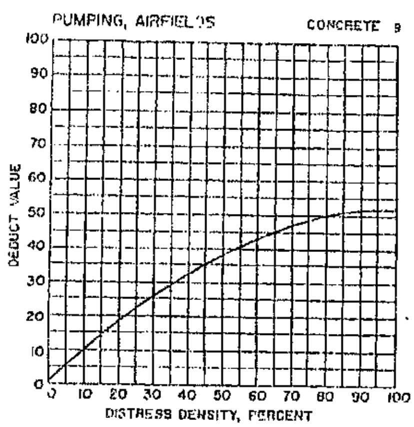


FIG. X4.9 Pumping

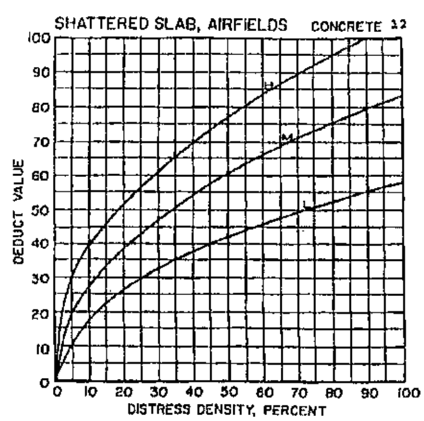


FIG. X4.12 Shattered Slab

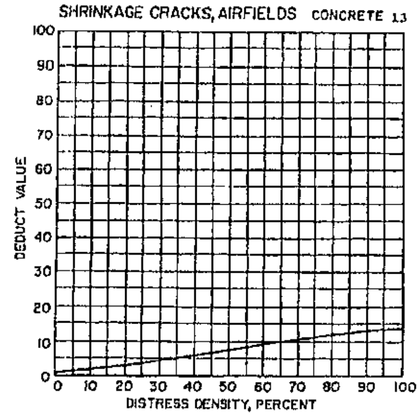


FIG. X4.13 Shrinkage Cracking

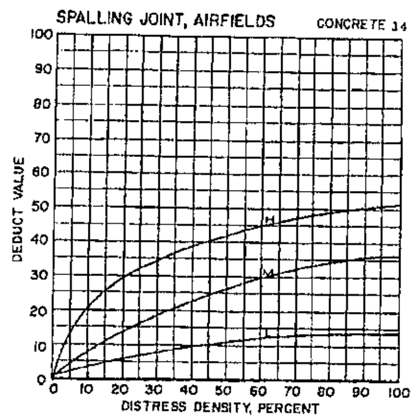


FIG. X4.14 Joint Spalling

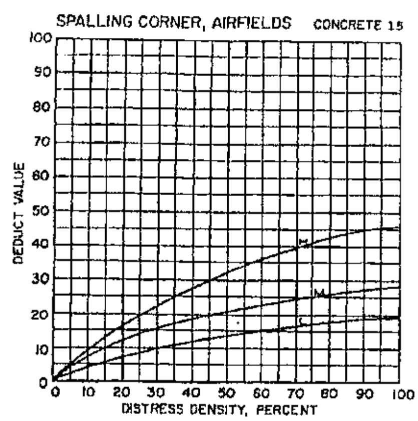


FIG. X4.15 Corner Spalling

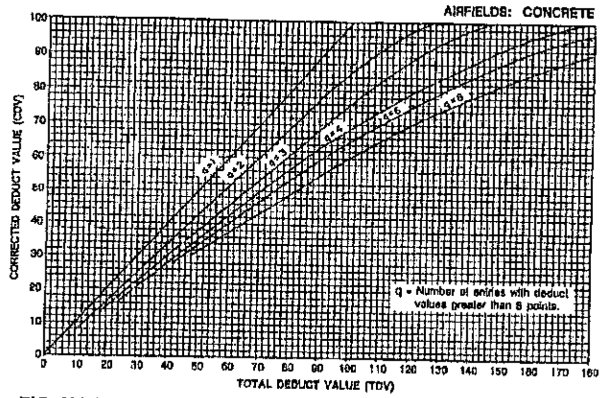


FIG. X4.16 Corrected DVs for Jointed Rigid Airfield Pavements

X5. BLANK FORMS

X5.1 See Figs. X5.1 and X5.2.

AIRFIELD ASPHALT PAVEMENT CONDITION SURVEY DATA SHEET FOR SAMPLE UNIT

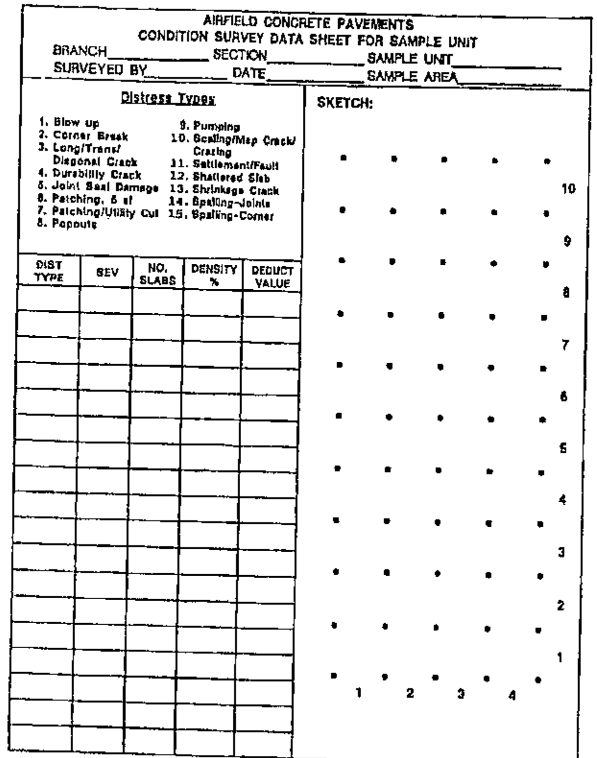
SKETCH:

BRANCH__________ SECTION________ SAMPLE UNIT________
SURVEYED BY__________ DATE________ SAMPLE AREA________

1. Alligator Cracking
2. Bleeding
3. Block Cracking
4. Corrugation
5. Depression
6. Jet Blast
7. Jt. Reflection (PCC)
8. Long. & Trans. Cracking
9. Oil Spillage
10. Patching
11. Polished Aggregate
12. Raveling/Weathering
13. Rutting
14. Shoving from PCC
15. Slippage Cracking
16. Swell

DISTRESS SEVERITY	QUANTITY									TOTAL	DENSITY %	DEDUCT VALUE

FIG. X5.1 Flexible Pavement Condition Survey Data Sheet for Sample Unit



AIRFIELD CONCRETE PAVEMENTS
CONDITION SURVEY DATA SHEET FOR SAMPLE UNIT

BRANCH________ SECTION________ SAMPLE UNIT________
SURVEYED BY________ DATE________ SAMPLE AREA________

Distress Types

1. Blow up
2. Corner Break
3. Long/Trans/ Diagonal Crack
4. Durability Crack
5. Joint Seal Damage
6. Patching, 5 sf
7. Patching/Utility Cut
8. Popouts
9. Pumping
10. Scaling/Map Crack/ Crazing
11. Settlement/Fault
12. Shattered Slab
13. Shrinkage Crack
14. Spalling-Joints
15. Spalling-Corner

DIST TYPE	SEV	NO. SLABS	DENSITY %	DEDUCT VALUE

SKETCH:

FIG. X5.2 Jointed Rigid Pavement Condition Survey Data Sheet for Sample Unit

REFERENCES

(1) Shahin, M. Y., Darter, M. I., and Kohn, S. D., "Development of a Pavement Maintenance Management System, Vol I, II, V," *Airfield Pavement Condition Rating*, US Air Force Civil Engineering Center, 1976.

(2) Kohn, S. D., and Shahin, M. Y., "Evolution of the Pavement Condition Index for Use on Porous Friction Surfaces," *Technical Report No. M-351*, US Army Construction Engineering Research Laboratory, Champaign, IL, 1984.

(3) Air Force Regulation 93-5, *Airfield Pavement Evaluation Program*, Department of the Air Force, Headquarters US Air Force, Washington, D.C.

(4) Advisory Circular No: 150/5380-6, *Guidelines and Procedures for Maintenance of Airport Pavements*, Federal Aviation Administration, US Department of Transportation.

(5) U.S. Naval Facilities Engineering Command Military Handbook 1021/2, "General Concepts for Airfield Pavement Design," 1988.

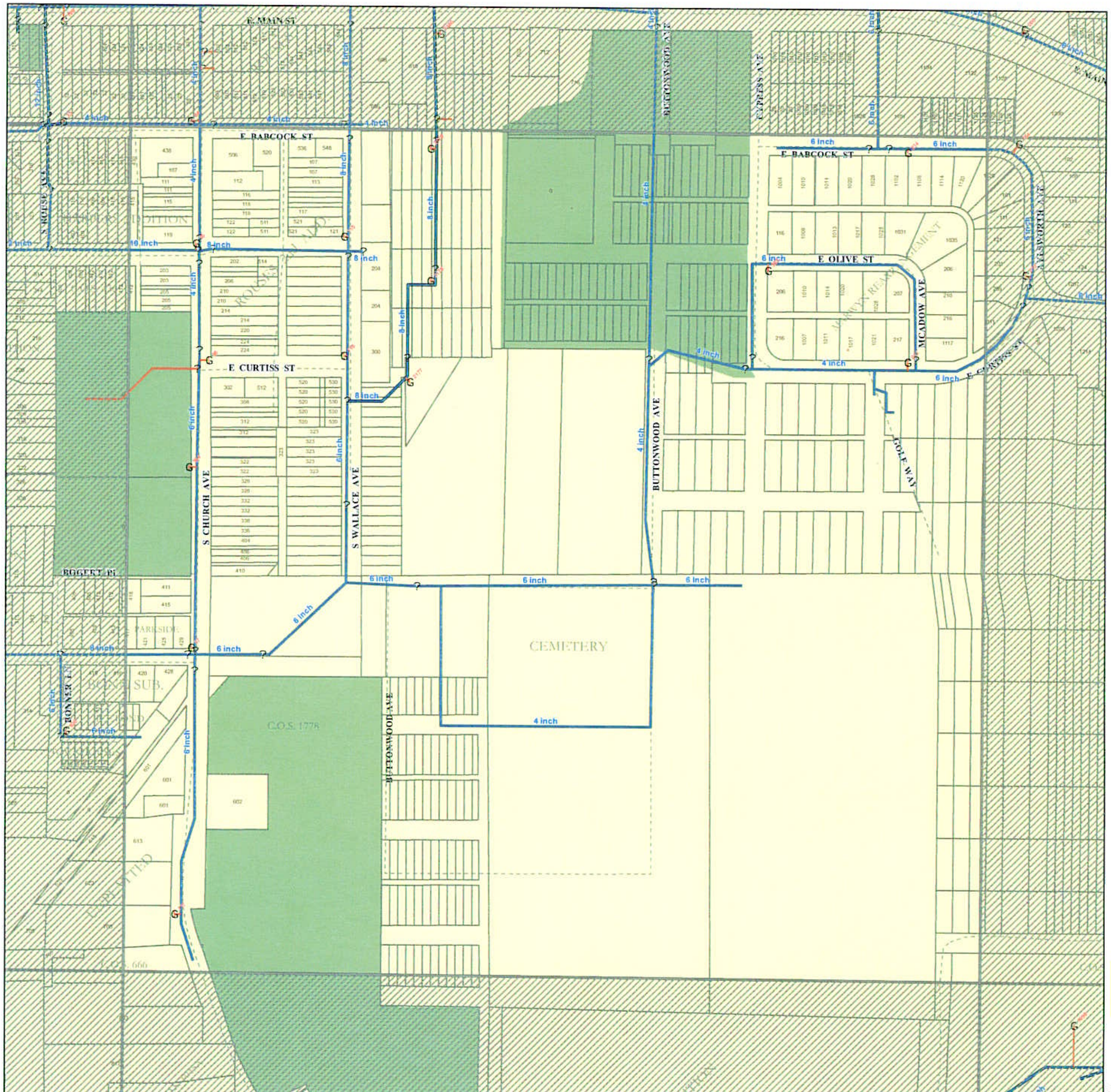
(6) *Pavement Condition Index (PCI) Field Manuals for Asphalt Surfaced Airfields*, American Public Works Association.

(7) *Pavement Condition Index (PCI) Field Manuals for Concrete Surfaced Airfields*, American Public Works Association.

(8) Green, W. H., and Eckrose, R. A., "Airport Pavement Inspection by PCI," 2nd edition, Eckrose/Green Associates, Madison, WI, 1988.

TETRA TECH

APPENDIX D:
City of Bozeman – Wallace Avenue Utility Locations



Legend

G Fire Hydrants

System Valves

- System Valve
- Fire Serivce
- Water Service

Control Valves (by type)

- Unknown
- Air Combination Valve
- Air Release Valve
- B Blowoff
- Bypass Valve
- Isolation Valve
- B PRV
- Hydrant Lateral Lines

Gravity Mains (by material)

- Cast Iron
- Concrete Cylinder Pipe
- Ductile Iron
- Steel
- PVC
- Parcels
- Roads
- Parks
- City Limits

F-4	E-4	D-4
F-5		D-5
F-6	E-6	D-6

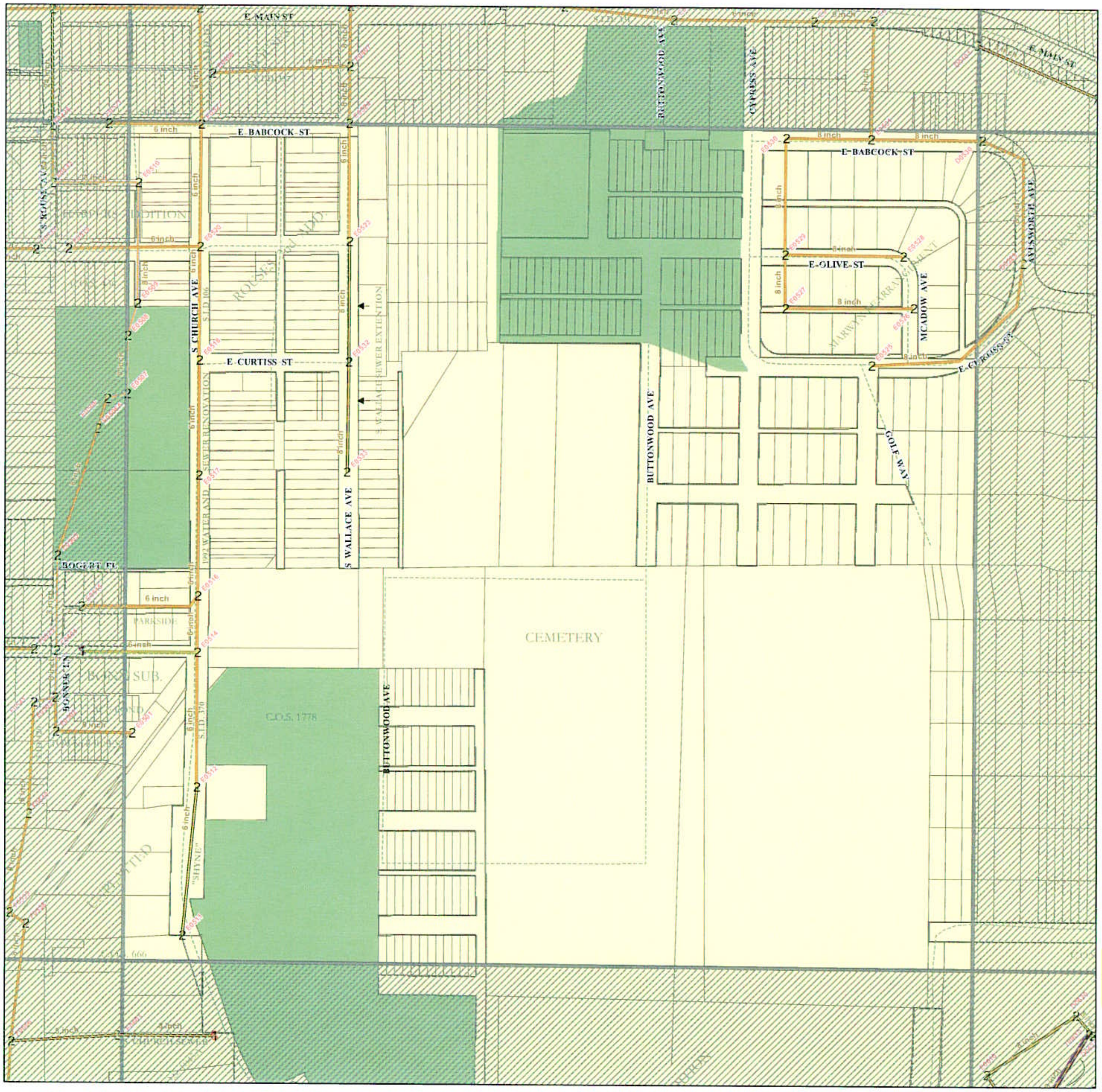
SE 1/4 Section 7 T2S R6E

E-5

Revised: Sep 12, 2007

1 inch equals 200 feet

200 100 0 200 Feet



Legend

- 2 Manholes
- Cleanouts
- Lampholes
- Caps
- R Taps
- Tees
- Wyes

Sewer Mains (by material)

- Asbestos Concrete
- Cast Iron
- Cement
- Clay
- Ductile Iron
- PVC
- Tile
- Vitrified Clay
- Slip Lines
- Parcels
- Roads
- Parks
- City Limits

F-4	E-4	D-4
F-5		D-5
F-6	E-6	D-6

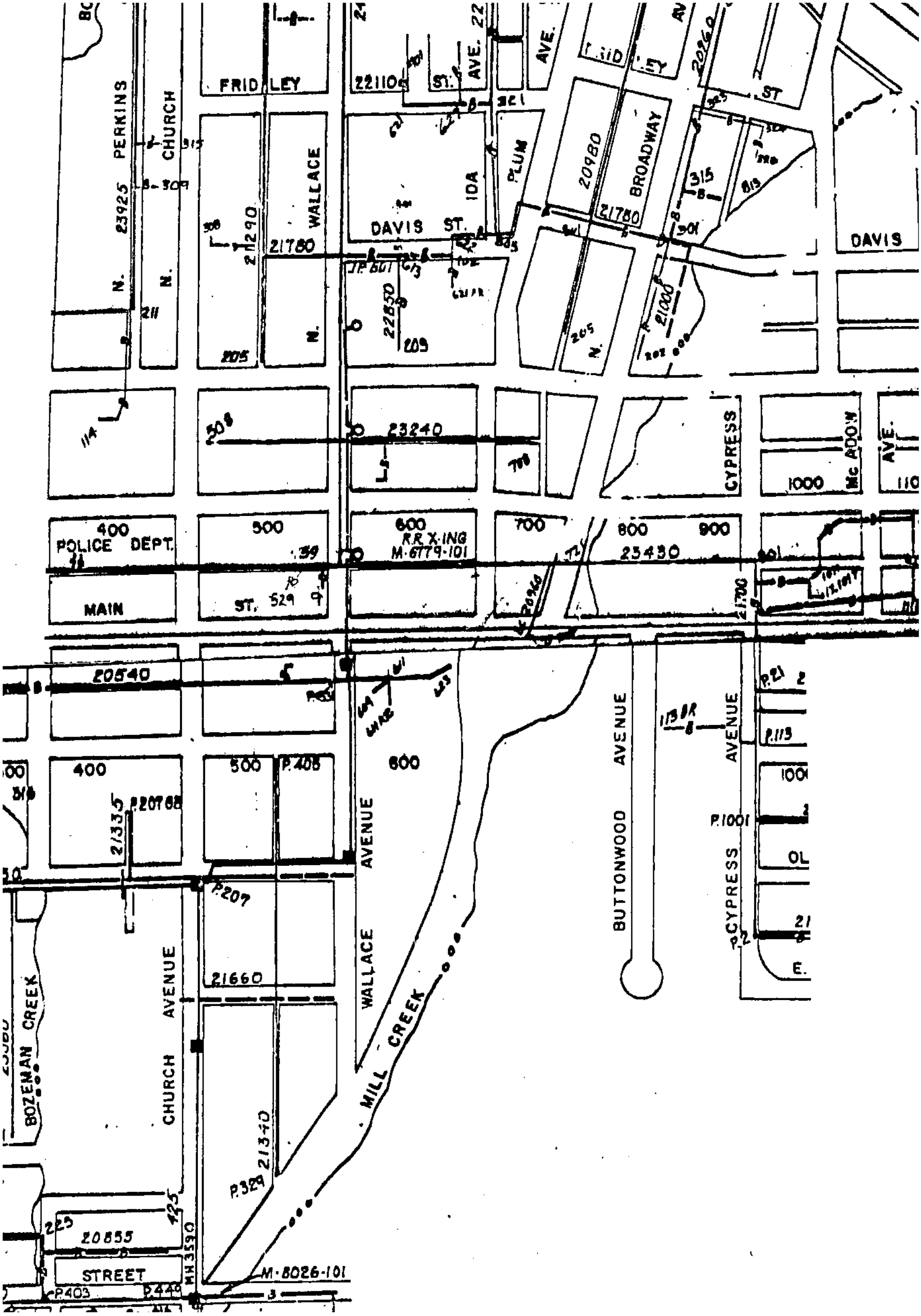
SE 1/4 Section 7 T2S R6E

E-5

Revised: Sep 03, 2007

1 inch equals 200 feet

200 100 0 200 Feet


FRIDLEY
FRIDLEY ST
22110
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AVE.
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PERKINS
CHURCH
N.
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WALLACE
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PLUM
BROADWAY
20980
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23925
21290
21780
DAVIS ST.
21780
DAVIS
22850
21000
ST
315
400
POLICE DEPT.
MAIN
500
ST.
600
R.R. X-ING
M-6779-101
700
800
900
23430
23240
CYPRESS
1000
Mc ADOW
AVE.
21700
20540
400
500
P.408
600
P.20768
21335
P.207
AVENUE
WALLACE
AVENUE
BUTTONWOOD
AVENUE
CYPRESS
AVENUE
P.113
P.1001
OL
E.
21660
CHURCH
AVENUE
BOZEMAN CREEK
MILL CREEK
21340
P.329
20855
STREET
MH 3590
M-8026-101
P.403
P.449

TETRA TECH

APPENDIX E:
Tetra Tech Test Pit Log

TETRA TECH

FIELD LOG OF EXPLORATION TEST PIT

JOB NO:______________________ PROJECT NAME:____

STATE:_________ COUNTY:_______________ LOGGED BY:____________________ TEST PIT NO.:__________________

DESCRIPTIVE LOCATION:____

DATE STARTED:_________________ DATE COMPLETED:_______________ EXCAVATION COMPANY___________

TOTAL DEPTH____

REMARKS:____

____

____

____

____

Depth (feet)	Classification and Description	Sample Depth (ft)	Headspace (ppm)

TETRA TECH

APPENDIX F:
CARB Method 435

State of California
AIR RESOURCES BOARD

Method 435

Determination of Asbestos Content of Serpentine Aggregate

Adopted: June 6, 1991

Method 435

Determination of Asbestos Content of Serpentine Aggregate

1.0Principle and Applicability

1.1Principle:

Asbestos fibers may be released from serpentine rock formations and are determined by microscopic techniques. The results are very sensitive to sampling procedures. The analytical results are reported in percent asbestos fibers which is the percent number of asbestos fibers contained in 400 randomly chosen particles of a bulk sample. Since the homogeneity of the material is unknown, the uncertainty in the sampling cannot be defined. The uncertainty of the analytical technique is two percent if twenty asbestos fibers are counted in a sample of 400 particles. The derivation of this uncertainty value is explained in Section 7.4.

1.2Applicability:

This method is applicable to determining asbestos content of serpentine aggregate in storage piles, on conveyor belts, and on surfaces such as roads, shoulders, and parking lots.

2.0Definitions:

2.1Bulk Sample - A sample of bulk material.

2.2Grab Sample - A sample taken from a volume of material.

2.3Composite Sample - A mixture or blend of material from more than one grab sample.

2.4Serpentine - Serpentinite, serpentine rock or serpentine material.

2.5Executive Officer - The term Executive Officer as used in this method shall mean the Executive Officer of the Air Resources Board (ARB) or Air Pollution Control Officer/Executive Officer of a local air pollution control district/air quality management district.

3.0 Applicable Sources:

This method can be used to obtain bulk material samples from three types of sources:

1. Serpentine aggregate storage piles.

2. Serpentine aggregate conveyor belts.

3. Serpentine aggregate covered surfaces.

4.0 Sampling Apparatus

4.1 Serpentine Aggregate Storage Piles

Tube insertion often provides the simplest method of aggregate material investigation and sampling. Insertion tubes shall be adequate to provide a relatively rapid continuous penetration force.

4.1.1 Thin-walled tubes should be manufactured as shown in Figure 1. The tube should have an outside diameter between 2 to 5 inches and be made of metal or plastic having adequate strength for penetration into aggregate piles. These tubes shall be clean and free of surface irregularities including projecting weld seams. Further information on these tubes can be found in Table 1 and ASTM D 1587-83, which is incorporated herein by reference.

4.1.2 The insertion tube can be made out of commercially available two inch PVC Schedule 40 pipe. Further information on the tube can be found in Table 2.

4.1.3 A round point shovel may be used.

4.2 Serpentine Aggregate Conveyor Belts

4.2.1 Sampling of aggregate off a conveyor belt requires a hand trowel, a small brush, and a dust pan.

4.2.2 Two templates as shown in Figure 2 are needed to isolate material on the conveyor belt.

4.2.3 An automated belt sampler may be used.

4.3Serpentine Aggregate Covered Surfaces

A shovel, a hand or machine-operated auger or other suitable equipment can be used to collect samples of aggregate materials on covered surfaces.

4.3.1Hand-Operated Augers

4.3.1.1Helical Augers-Small lightweight augers such as spiral-type augers and ship-type augers may be used. A description of these augers types can be found in ASTM D 1452-80, which is incorporated herein by reference.

4.3.1.2Orchard barrel and open spiral type tubular augers may be used to collect samples. These augers range in size from 1.5 through 8 inches, and have the common characteristic of appearing essentially tubular when viewed from the digging end. Further description of these auger types can be found in ASTM D 1452-80.

4.3.1.3Clam Shell or Iwan Type post-hole augers may be used to collect samples from surfaces generally 2 through 8 inches in diameter and have a common mean of blocking the escape of soil from the auger. Further description of these augers can be found in ASTM D 1452-80.

4.3.2Machine-Operated Augers

Machine-Operated Augers such as helical augers and stinger augers may be used. These augers are normally operated by heavy-duty, high-torque machines, designed for heavy construction work. Further description of these augers can be found in ASTM D 1452-80.

4.3.3A round point shovel can also be used to obtain a sample of aggregate covered surface material.

5.0Sampling

The sampling procedure has been developed to provide an unbiased collection of bulk samples. A sampling plan, including a description of how the grab samples will be randomly collected and the number of samples to be collected, shall be developed. Prior to conducting any sampling the sampling plan shall be submitted to the Executive Officer for approval, if the sampling is conducted for determining compliance with a rule or regulation. The amount of composite 200 mesh material, as described below, shall be sufficient to provide a sample to the source or Executive Officer, if requested, and a sample to be archived for future use.

A single test as described below shall cover:

a) 1000 tons of aggregate for piles and conveyor belts, or
b) one acre aggregate covered surface, or
c) one mile of aggregate covered road, or
d) two acres or two miles of dual aggregate covered shoulders.

Exposure to airborne asbestos fibers is a health hazard. Asbestos has been listed by the Governor as causing cancer and identified by the Air Resources Board as a toxic air contaminant. Serpentine aggregate may contain asbestos. Bulk samples collected can contain friable asbestos fibers and may release fibers during sampling, handling or crushing steps. Adequate safety precautions should be followed to minimize the inhalation of asbestos fibers. Crushing should be carried out in a ventilated hood with continuous airflow (negative pressure) exhausting through an HEPA filter. Handling of samples without these precautions may result in the inhalation of airborne asbestos fibers.

5.1 Serpentine Aggregate Storage Piles

Serpentine aggregate storage piles typically have a conical or a triangular prism shape. The aggregate is introduced at the top of the pile and is allowed to flow over the side. This action, called sloughing, causes a size segregation to occur with the finer material deposited towards the top of the pile.

The locations where grab samples will be taken are randomly chosen over the surface of the pile. The method of randomly choosing the sampling locations is left up to sampling personnel but must follow the procedures specified in the sampling plan. For 1000 tons of product, a grab sample shall be taken at a minimum of three randomly chosen sampling locations. A minimum of three grab samples shall be taken even if the product pile contains less than 1000 tons of material. The slough is raked or shoveled away from the sampling location. A sampling apparatus is inserted one foot into the pile and the material is removed and is placed in an appropriate sized sampling container. Some of the possible sampling apparatus is discussed in Section 4.1. Each of the grab samples shall be placed in the same sample container. This composited sample shall be crushed to produce a material with a nominal size of less than three-eighths of an inch. Before crushing, the sample must be adequately dried. ASTM Method C-702-80, which is incorporated herein by reference, shall be used to reduce the size of the crushed grab sample to a one pint aliquot. The one pint aliquot, shall be further crushed using a Braun mill or equivalent to produce a material of which the majority shall be less than 200 Tyler mesh. An aliquot of the 200 mesh material shall be put into a labeled sealed container. The label shall contain all the information described in Section 6 (except item 4).

5.2Serpentine Aggregate Conveyor Belts

Serpentine aggregate is transported from the rock crushing plant to a product stacking belt and finally to a storage pile or to a waiting truck for delivery to a buyer.

The grab samples shall be taken from the product stacking belt or if this is not possible then at the first transfer point before the stockpile. The grab samples shall be collected by stopping the belt a minimum of three times or using an automated sampler. The method of randomly choosing the sampling locations and intervals is left up to sampling personnel but must follow the procedure specified in the sampling plan. For 1000 tons of product, a grab sample is taken at a minimum of three randomly selected intervals. A minimum of three samples shall be taken even if the generated product is less than 1000 tons. Each time the belt is stopped to take a grab sample, templates, as shown in Figure 2, are placed a minimum of six inches apart to isolate the material on the belt. The material within the templates is removed with a small shovel or with a brush and dust pan for the finer material and is placed in an appropriate sized sampling container. Each of the grab samples shall be placed in the same sample container. This composited sample shall be crushed to produce a material with a nominal size of less than three eighths of an inch. Before crushing, the sample must be adequately dried. ASTM Method C-702-80, which is incorporated herein by reference, shall be used to reduce the size of the crushed grab sample to an one pint aliquot. The one pint aliquot shall be further crushed using a Braun mill or equivalent to produce a material which the majority of which shall be less than 200 Tyler mesh. An aliquot of the 200 mesh material shall be put into a labeled sealed container. The label must contain all the information listed in Section 6 (except item 4).

5.3 Serpentine Aggregate Covered Surfaces.

5.3.1Serpentine Aggregate Covered Roads

A serpentine aggregate-covered road shall be characterized by taking grab samples from a minimum of three randomly chosen locations per mile of road. The method of randomly choosing the sampling locations is left up to sampling personnel but must follow the procedures specified in the sampling plan. A minimum of three samples shall be taken even if the road is less than one mile long. Section 4.3 describes some of the possible sampling apparatus used to collect the grab samples. Grab samples shall not contain underlying soils. Each of the grab samples shall be placed in the same sample container. This composited sample shall be crushed to produce material with a nominal size less than three-eighths of an inch. Before crushing, the sample must be adequately dried. ASTM Method C-702-80 shall be used to reduce the size of the crushed grab sample to an one pint aliquot. The one pint aliquot shall be further crushed using a Braun mill or equivalent to produce a material which the majority shall be less than 200 Tyler mesh. An aliquot of the 200 mesh material shall be put into a labeled sealed container. The label must contain all the information listed in Section 6 (except item 4).

5.3.2Serpentine Aggregate Covered Areas

A serpentine aggregate-covered play yard or parking lot shall be characterized by taking grab samples from a minimum of three randomly chosen locations per acre. The method of randomly choosing the sampling locations is left up to sampling personnel but must follow the procedure specified in the sampling plan. A minimum of three samples shall be taken even if the area is less than one acre. Section 4.3 describes some of the possible sampling apparatus for collecting the sample. Grab samples shall not contain underlying soils. Each of the grab samples shall be placed in the same sample container. This composited sample shall be crushed to produce a material with a nominal size of less than three-eighths of an inch. Before crushing, the sample must be adequately dried. ASTM Method C-702-80 shall be used to reduce the size of the crushed grab sample to a one pint aliquot. The one pint aliquot shall be further crushed using a Braun mill or equivalent to produce a material which the majority shall be less than 200 Tyler mesh. An aliquot of the 200 mesh material shall be put into a labeled sealed container. The label must contain all the information listed in Section 6 (except item 4).

5.3.3Serpentine Aggregate Covered Road Shoulders

The sampling procedure specified in Section 5.3.1 or 5.3.2 shall be used for road shoulders covered with serpentine aggregate. The only difference is that a minimum of three grab samples shall be taken over a length of two miles of shoulder or over an area of two acres of shoulder surface. The word shoulder is meant to imply shoulders on both sides of the road. For serpentine aggregate covered shoulders, the sampling plan specified in Section 5 shall indicate whether the samples are collected on a two mile or two acre basis.

6.0Sampling Log

A sample log must be kept showing:

1. A unique sample number.
2. Facility name.
3. Facility address or location where sample is taken.

4. A rough sketch, video tape, or photograph of the specific sampling locations.
5. Date and time of sampling.
6. Name of person performing sampling.

7.0 Analytical Procedure

7.1Principle and Applicability

Samples of serpentine aggregate taken for asbestos identification are first examined for homogeneity and preliminary fiber identification at low magnification. Positive identification of suspect fibers is made by analysis of subsamples with the polarized light microscope.

The principles of optical mineralogy are well established.[2,3] A light microscope equipped with two polarizing filters coupled with dispersion staining is used to observe specific optical characteristics of a sample. The use of plane polarized light allows the determination of refractive indices along specific crystallographic axes. Morphology and color are also observed. A retardation plate is placed in the polarized light path for determination of the sign of elongation using orthoscopic illumination. Orientation of the two filters such that their vibration planes are perpendicular (crossed polars) allows observation of the birefringence and extinction characteristics of anisotropic particles.

Quantitative analysis involves the use of point counting. Point counting is a standard technique in petrography for determining the relative areas occupied by separate minerals in thin sections of rock. Background information on the use of point counting[3] and the interpretation of point count data[4] is available.

This method is applicable to all bulk samples of serpentine aggregate submitted for identification and quantification of asbestos components.

7.2Range

The analytical method may be used for analysis of samples containing from 0 to 100 percent asbestos. The upper detection limit is 100 percent. The lower detection limit is 0.25 percent.

7.3Interferences

Fibrous organic and inorganic constituents of bulk samples may interfere with the identification and quantification of the asbestos content. Fine particles of other materials may also adhere to fibers to an extent sufficient to cause confusion in the identification.

7.4Analytical Uncertainty

The uncertainty of the analytical method is two percent if twenty asbestos fibers are counted in a sample of 400 particles. The uncertainty of the analytical method may be assessed by a 95% confidence interval for the true percentage of asbestos fibers in the rock. The number of asbestos fibers in the sample is assumed to have a binomial distribution. If twenty asbestos fibers are found in a sample of 400 particles, a one-sided confidence interval for the true percentage has an upper bound of seven percent or an analytical uncertainty of two percent[11]. The confidence interval used here is an "exact" interval computed directly from the binomial distribution.

7.5Apparatus

7.5.1 Microscope

A low-power binocular microscope, preferably stereoscopic, is used to examine the bulk sample as received.

* Microscope: binocular, 10-45X

* Light Source: incandescent, fluorescent, halogen or fiber optic

* Forceps, Dissecting Needles, and Probes

* Glassine Paper, Clean Glass Plate, or Petri dish

* Compound microscope requirements: A polarized light microscope complete with polarizer, analyzer, port for wave retardation plate, 360° graduated rotating stage, substage condenser, lamp, and lamp iris.

* Polarized Light Microscope: described above

* Objective Lenses: 10X

* Dispersion Staining Objective Lens: 10X

* Ocular Lens:10X

* Eyepiece Reticule: 25 point or 100 point Chalkley Point Array or cross-hair

* Compensator Plate: 550 millimicron retardation

* First Order Red I Compensator, 530 nanometers

7.6Reagents

Refractive Index Liquids: 1.490-1.570, 1.590-1.720 in increments of 0.002 or 0.004.

Refractive Index Liquids for Dispersion Staining: High-dispersion series, 1.550,1.605,1.630 (optional)

UICC Asbestos Reference Sample Set: Available from UICC MRC Pneumoconiosis Unit, Lisndough Hospital Penarth, Glamorgan CF6 1xw, UK and commercial distributors.

Tremolite-asbestos: Available from J. T. Baker

Actinolite-asbestos: Available from J. T. Baker

Chrysotile, Amosite, and Crocidolite is available from the National Institute of Standards and Technology.

Anthophyllite, Tremolite, Actinolite will be available from the National Institute of Standards and Technology during the first quarter of 1990.

8.0Procedures

Exposure to airborne asbestos fibers is a health hazard. Bulk samples submitted for analysis are usually friable and may release fibers during handling or matrix reduction steps. All samples and slide preparations should be carried out in a ventilated hood or glove box with continuous airflow (negative pressure) exhausting through an HEPA filter. Handling of samples without these precautions may result in exposure of the analyst and contamination of samples by airborne fibers.

8.1Sample Preparation

An aliquot of bulk material is removed from the one pint sample container. The aliquot is spread out on a glass slide. A drop of staining solution with appropriate refractive index is added to the aliquot. A cover slide is placed on top of the sample slide.

The first preparation should use the refractive index solution for Chrysotile. If during the identification phase other asbestiforms are suspected to be present in the sample, due to their morphology, then additional analyses shall be performed with the appropriate solutions. Report the percentages of each asbestiform and combine percentages to determine total asbestos concentrations.

8.2Fiber Identification

Positive identification of asbestos requires the determination of the following optical properties:

Morphology (3 to 1 minimum aspect ratio)
Color and pleochroism
Refractive indices
Birefringence
Extinction characteristics
Sign of elongation

Table 3 lists the above properties for commercial asbestos fibers. Natural variations in the conditions under which deposits of asbestiform minerals are formed will occasionally produce exceptions to the published values and differences from the UICC standards. The sign of elongation is determined by use of the compensator plate and crossed polars. Refractive indices may be determined by the Becke line test. Becke line test or dispersion staining shall be used to identify asbestos fibers. Central stop dispersion staining colors are presented in Table 4. Available high-dispersion (HD) liquids should be used.

8.3 Quantification of Asbestos Content

Asbestos quantification is performed by a point-counting procedure. An ocular reticle (point array) or cross-hair is used to visually superimpose points on the microscope field of view. The point counting rules are as follows:

1. Record the number of points positioned directly above each particle or fiber.

2. Record only one point if two points are positioned over same particle or fiber.

3. Record the number of points positioned on the edge of a particle or fiber.

4. If an asbestos fiber and a matrix particle overlap so that a point is superimposed on their visual intersection, a point is scored for both categories.

5. If a test point lies over an ambiguous structure, no particle or fiber is recorded. Examples of "ambiguous" structures are:

    a. fibers whose dispersion colors are difficult to see

    b. structures too small to categorize

6. A fiber mat or bundle is counted as one fiber.

For the purpose of the method, "asbestos fibers" are defined as mineral fibers having an aspect ratio greater than 3:1 and being positively identified as one of the minerals in Table 3.

A total of 400 points superimposed on either asbestos fibers or nonasbestos matrix material must be counted over at least eight different preparations of representative subsamples. Take eight forceps samples and mount each separately with the appropriate refractive index liquid. The preparation should not be heavily loaded. The sample should be uniformly dispersed to avoid overlapping particles and allow 25-50 percent empty area within the fields of view. Count 50 nonempty points on each preparation, using either

A reticle with 100 points (Chalkley Point Array) and counting 25 points in at least two randomly selected fields.

or

A reticle with 25 points (Chalkley Point Array) and counting at least two randomly selected fields.

or

A reticle with a standard cross-hair and counting at least 50 randomly selected fields.

For samples with mixtures of isotropic and anisotropic materials present, viewing the sample with slightly uncrossed polars or the addition of the compensator plate to the polarized light path will allow simultaneous discrimination of both particle types. Quantitation should be performed at 100X. Confirmation of the quantitation result by a second analyst on 10 percent of the analyzed samples should be used as standard quality control procedure. All optical properties in Section 8.2 shall be determined to positively identify asbestos.

EXCEPTION I

If the sample is suspected of containing no asbestos a visual technique can be used to report that the sample does not contain asbestos. The rules are as follows:

1. Prepare three slides as described in Section 8.3.
2. View 10 fields per preparation. Identify all fibers.
3. If all fibers are nonasbestos, report no asbestos were found and that the visual technique was used.

4. If one fiber is determined to be asbestos, discontinue the visual method and perform the point counting technique as described above.

EXCEPTION II

If the sample is suspected to have an asbestos content in excess of ten percent, a visual technique can be used to report that the sample contains greater than ten percent asbestos. The standard operating procedure of the visual technique allowed in the National Institute of Standards and Technology's National Voluntary Laboratory Accreditation Program, Bulk Asbestos Handbook, National Institute of Standards and Technology publication number NISTIR 88-3879 dated October 1988, which is incorporated herein by reference, shall be followed.

9.0Calculations

The percent asbestos is calculated as follows:

% asbestos = (a/n) 100% where
a = number of asbestos counts,
n = number of nonempty points counted(400)
If a = 0, report "No asbestos detected"
If a > 0, report the calculated value to the nearest 0.25% percent

If "no asbestos detected" is reported by the point counting technique, the analyst may report the observation of asbestos fibers in the non-counted portions of the sample.

10.0Alternative Methods

10.1Alternative Sampling Methods

Alternate sampling methods may be used as long as they are substantially equivalent to the sampling methods discussed in Section 5 and approved by the Executive Officer of the Air Resources Board. The ARB Executive Officer may require the submittal of test data or other information to demonstrate equivalency.

10.2Analytical Methods

An alternative analytical method may be used as long as it produces results substantially equivalent to the results produced by the point counting method and approved by the Executive Officer of the Air Resources Board. The ARB Executive Officer may require the submittal of test data or other information to demonstrate equivalency.

11.0References

1. G. S. Koch, Jr., R. F. Link, Statistical Analysis of Geological Data, New York, Dover Publications, Inc., December 1985.

2. Paul F. Kerr, Optical Mineralogy, 4th ed., New York, McGraw-Hill, 1977.

3. E. M. Chamot and C. W. Mason, Handbook of Chemical Microscopy, Volume One, 3rd ed., New York: John Wiley & Sons, 1958

4. F. Chayes, Petrographic Model Analysis: An Elementary Statistical Appraisal, New York: John Wiley & Sons, 1958.

5. E.P. Brantly, Jr., K. W. Gold, I. E. Myers, and D. E. Lentzen, Bulk Sample Analysis for Asbestos Content: Evaluation of the Tentative Method, U.S. Environmental Protection Agency, October 1981.

6. U.S. Environmental Protection Agency, Asbestos-Containing Materials in School Buildings: A Guidance Document, Parts 1 and 2 EPA/OTS No. C00090m Narcg 1979.

7. D. Lucas, T. Harwell, and A. V. Rao, Asbestos Containing Materials in School Buildings: Guidance for Asbestos Analytical Programs, EPA 580/13-80-017a, U. S. Environmental Protection Agency, December 1980.

8. D.H. Taylor and J.S. Bloom, Hexametaphosphate Pretreatment of Insulation Samples For Identification of Fibrous Constituents, Microscope, 28, 1980.

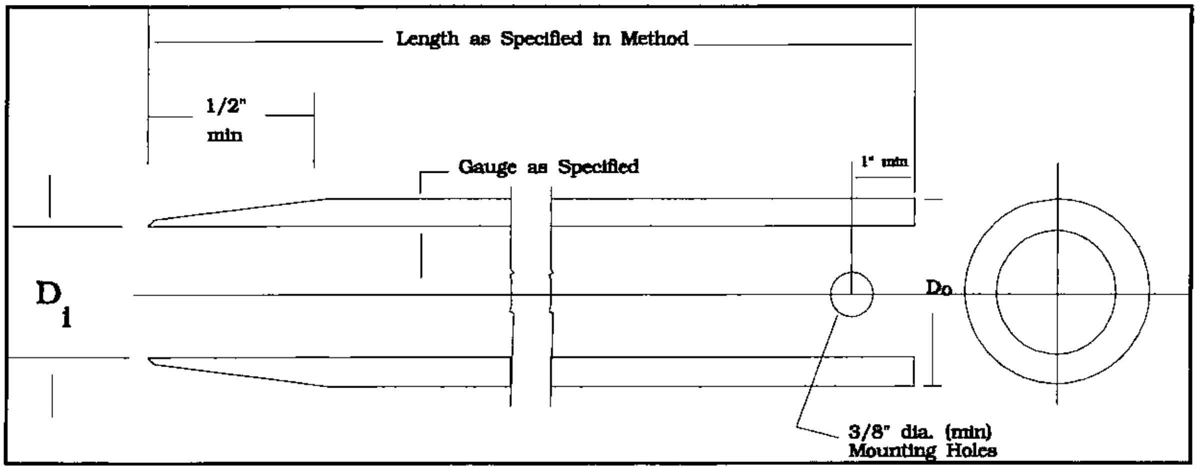
9. W. J. Campbell, R. L. Blake, L. L. Brown, E. E. Cather, and J. J. Sjoberg. Selected Silicate Minerals and Their Asbestiform Varieties: Mineralogical Definitions and Identification-Characterization, U. S. Bureau of Mines Information Circular 8751, 1977.

10. Walter C. McCrone, Asbestos Particle Atlas, Ann Arbor, Ann Arbor Science Publishers, June 1980.

11. John Moore, Biostatistician, Personnel Communication, February 8, 1990.

Figure 1

Thin Wall Tube for Sampling



Note 1 Minimum of two mounting holes on opposite sides for 2 to 3 inch diameter sampler.

Note 2 Minimum of four mounting holes spaced at 90° for samplers 4 inch diameter and larger.

Note 3 Tube held with hardened screws.

Note 4Two inch outside-diameter tubes are specified with an 18-gauge wall thickness to comply with area ratio criteria accepted for "undisturbed samples." Users are advised that such tubing is difficult to locate and can be extremely expensive in small quantities. Sixteen-gauge tubes are generally readily available.

Table 1

Suitable Thin Walled Steel Sample Tube[A]

Outside diameter:			
inches	2	3	5
millimeters	50.8	76.2	127
Wall thickness:			
Bwg	18	16	11
inches	0.049	0.065	0.120
millimeters	1.24	1.65	3.05
Tube length:			
inches	36	36	54
meters	0.91	0.91	1.45
Clearance ratio, %	1	1	1

[A] The three diameters recommended in Table 1 are indicated for purposes of standardization, and are not intended to indicate that sampling tubes of intermediate or larger diameters are not acceptable. Lengths of tubes shown are illustrative. Proper lengths to be determined as suited to field conditions.

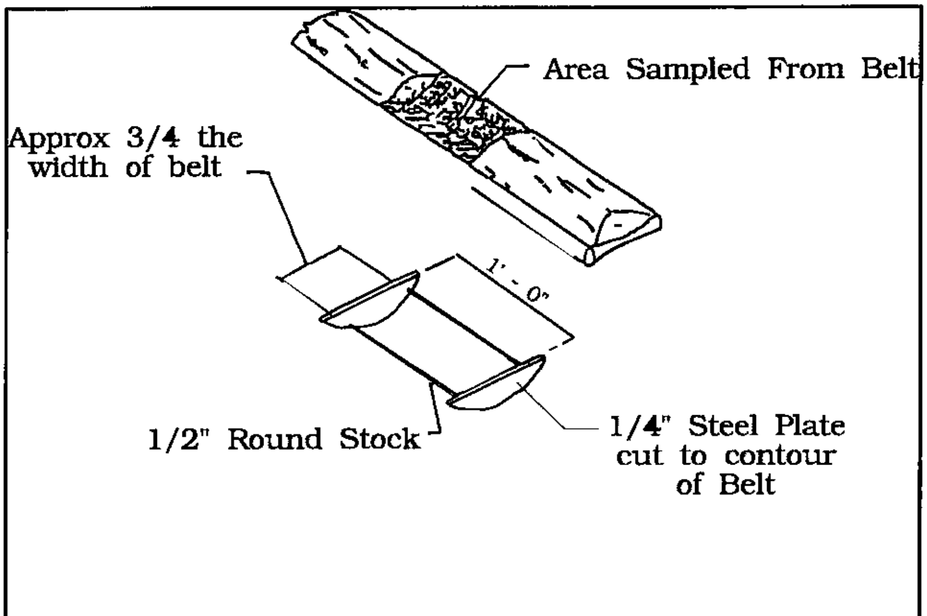
Table 2

Dimensional Tolerances for Thin Walled Tubes

Nominal Tube Diameters from Table 1[A]	Tolerances, inches		
Size Outside Diameter	2	3	4
Outside diameter	+0.007 -0.000	+0.010 -0.000	+0.015 -0.000
Inside diameter	+0.000 -0.007	+0.000 -0.010	+0.000 -0.015
Wall thickness	+0.007	+0.010	+0.015
Ovality	0.015	0.020	0.030
Straightness	0.030/ft	0.030/ft	0.030/ft

[A]Intermediate or larger diameters should be proportional. Tolerances shown are essentially standard commercial manufacturing tolerances for seamless steel mechanical tubing. Specify only two of the first three tolerances; O.D. and I.D. or O.D. and Wall, or I.D. and Wall.

Figure 2



Typical Template to Drag Sample Off Belt

Table 3
Optical Properties of Asbestos Fibers

Mineral	Morphology, color	Refractive Indices[b] alpha	Refractive Indices[b] gamma	Birefringence	Extinction	Sign of Elongation
Chrysotile fiber (asbestiform serpentine)	Wavy fibers. fiber bundles have splayed ends and "kinks" Aspect ratio typically > 10:1. Colorless, nonpleochroic.	1.493-1.560	1.517-1.562[f] (normally 1.556)	.002-.014	|| to length	+ (length slow)
Amosite fiber (asbestiform grunerite)	Straight, rigid fibers. Aspect ratio typically >10:1. Colorless to brown, nonpleochroic or weakly so. Opaque inclusions may be present.	1.635-1.696	1.655-1.729[f] (normally 1.696-1.710)	.020-0.33	|| to length	+ (length slow)
Crocidolite fiber (asbestiform riebeckite)	Straight, rigid fibers. Thick fibers and bundles common, blue to purple-blue in color. Pleochroic. Birefringence is generally masked by blue color.	1.654-1.701	1.668-1.717 (normally close to 1.700	.014-0.016	|| to length	- (length fast)
Anthophyllite-asbestos fiber	Straight fibers and fiber bundles showing splayed ends. Colorless to light brown. Pleochroism absent.	1.596-1.652	1.615-1.676[f]	.019-.024	|| to length	+ (length slow)

Tremolite-actinolite-asbestos	+ (length slow) Straight and curved fibers and fiber bundles. Large bundles show splayed ends. Tremolite is Colorless and actinolite is green weakly to moderately pleochroic.	1.599-1.668	1.622-1.688[f]	.023-.020	|| to length

a From References 6; colors cited are seen by observation with plane polarized light.
b From References 7 and 9.
c Fibers subjected to heating may be brownish.
d fibers defined as having aspect ratio >3:1.
e to fiber length.
f || to fiber length.

Table 4

Central Stop Dispersion Staining Colors[a]

Mineral	RI Liquid	nu |	nu ||
Chrysotile	1.550HD	Blue	blue-magenta
Amosite	1.680	blue-magenta to pale blue	Golden-Yellow
	1.550HD	Yellow to white	Yellow to white
Crocidolite[b]	1.700	Red magenta	Blue-magenta
	1.550HD	Yellow to white	Yellow to white
Anthophyllite	1.605HD	Blue	Gold to Gold-magenta
Tremolite	1.605HD[c]	Pale blue	Yellow
Actinolite	1.630HD	Gold-magenta to blue	Gold
	1.630HD[c]	Magenta	Golden-yellow

a From Reference 10.
b Blue absorption color.
c Oblique extinction view.

APPENDIX G:
EPA Method 600/R-93/116

United States
Environmental Protection
Agency

Office of Research and
Development
Washington, DC 20460

EPA/600/R-93/116
July 1993

EPA

Test Method

Method for the Determination of Asbestos in Bulk Building Materials

FP D-2 missing in Side -

TECHNICAL REPORT DATA

1. REPORT NO.	2.	3. RECIPIENT'S ACCESSION NO.
EPA/600/R-93/116		PB93-218576

4. TITLE AND SUBTITLE	5. REPORT DATE
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15. SUPPLEMENTARY NOTES

Although this method has been developed as a candidate for compliance monitoring for EPA programs, it must be officially designated by a program office in their regulations before it can be used for compliance monitoring. The user should verify the official status of the method before using it for compliance monitoring purposes.

16. ABSTRACT

A method describing the qualitative and quantitative analysis of bulk building materials for asbestos content is described. The method employs polarized light microscopy (PLM), x-ray diffraction (XRD), and analytical transmission electron microscopy (AEM) for qualitative identification of materials. Quantitative analysis is accomplished by comparison of gravimetrically prepared standards of known composition with unknown samples using a combination of visual comparison, point counting, gravimetry and quantitative x-ray diffraction.

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TEST METHOD

METHOD FOR THE DETERMINATION OF ASBESTOS IN BULK BUILDING MATERIALS

by

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DISCLAIMER

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TABLES

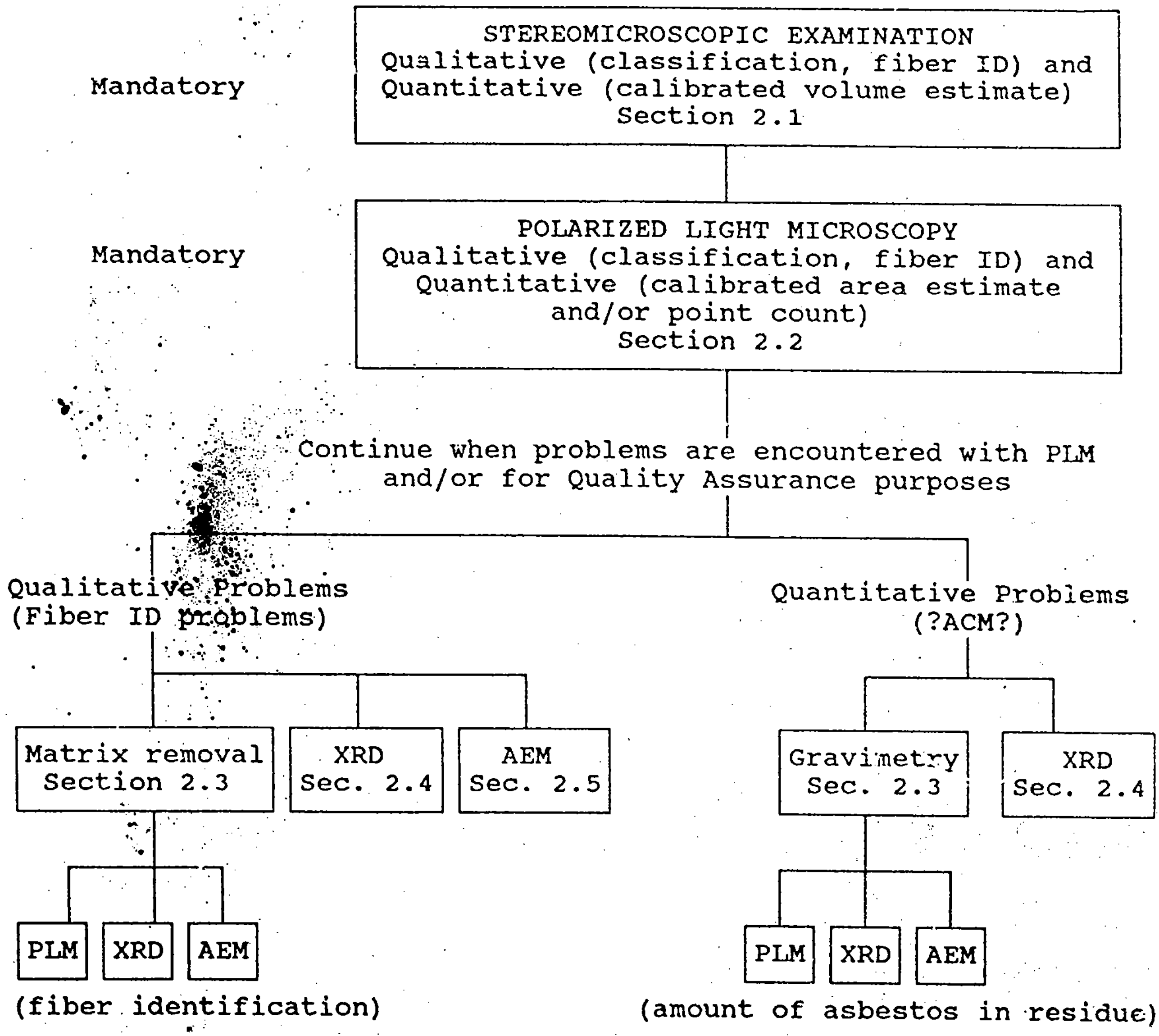
1.0 INTRODUCTION

Laboratories are now called upon to identify asbestos in a variety of bulk building materials, including loose-fill insulations, acoustic and thermal sprays, pipe and boiler wraps, plasters, paints, flooring products, roofing materials and cementitious products.

The diversity of bulk materials necessitates the use of several different methods of sample preparation and analysis. An analysis with a simple stereomicroscope is always followed by a polarized light microscopic (PLM) analysis. The results of these analyses are generally sufficient for identification and quantitation of major concentrations of asbestos. However, during these stereomicroscopic and PLM analyses, it may be found that additional techniques are needed to: 1) attain a positive identification of asbestos; 2) attain a reasonable accuracy for the quantity of asbestos in the sample; or 3) perform quality assurance activities to characterize a laboratory's performance. The additional techniques include x-ray diffraction (XRD), analytical electron microscopy (AEM), and gravimetry, for which there are sections included in the method. Other techniques will be considered by the Environmental Protection Agency (EPA) and may be added at some future time. Table 1-1 presents a simplified flowchart for analysis of bulk materials.

This Method for the Determination of Asbestos in Bulk Building Materials outlines the applicability of the various preparation and analysis methods to the broad spectrum of bulk building materials now being analyzed. This method has been evaluated by the EPA Atmospheric Research and Exposure Assessment Laboratory (EPA/AREAL) to determine if it offers improvements to current analytical techniques for building materials. This method demonstrated a capability for improving the precision and accuracy of analytical results. It contains significant revisions to procedures outlined in the Interim Method,[1] along with the addition of several new procedures. Each technique may reduce or introduce bias, or have some effect on the precision of the measurement, therefore results need to be interpreted judiciously. Data on each technique, especially those new to asbestos analysis, will be collected over time and carefully evaluated, with resulting recommendations for changes to the Method to be passed on to the appropriate program office within EPA.

TABLE 1-1. SIMPLIFIED FLOWCHART FOR ANALYSIS OF BULK MATERIALS



This is an analytical method. It is not intended to cover bulk material sampling, an area addressed previously[2,3,4,5] by the EPA. However, subsampling or sample splitting as it pertains to laboratory analysis procedures in this method, is discussed throughout.

1.1 References

1. **Interim Method for the Determination** of Asbestos in Bulk Insulation Samples, U.S. E.P.A. 600/M4-82-020, 1982.
2. **Asbestos-Containing Materials in School Buildings:** A Guidance Document, Part 1 and 2, U.S. E.P.A./O.T.S NO. C00090, 1979.
3. **Asbestos in Buildings:** Simplified Sampling Scheme for Friable Surfacing **Materials**, U.S. E.P.A. 560/5-85-030a, 1985.
4. **Guidance for Controlling** Asbestos-Containing Materials in Buildings, U.S. E.P.A. 560/5-85-024, 1985.
5. **Asbestos-Containing Materials in Schools:** Final Rule and Notice, 40 CFR Part 763, October, 1987.

2.0 METHODS

2.1 Stereomicroscopic Examination

A preliminary visual examination using a simple stereomicroscope is <u>mandatory</u> for all samples. A sample should be of sufficient size to provide for an adequate examination. For many samples, observations on homogeneity, preliminary fiber identification and semi-quantitation of constituents can be made at this point. Another method of identification and semi-quantitation of asbestos <u>must be</u> used in conjunction with the stereomicroscopic examination. A description of the suggested apparatus needed for stereomicroscopic examination is given in Appendix B.

The laboratory should note any samples of insufficient volume. A sufficient sample volume is sample-type dependent. For samples such as floor tiles, roofing felts, paper insulation, etc., three to four square inches of the layered material would be a preferred sample size. For materials such as ceiling tiles, loose-fill insulation, pipe insulation, etc., a sample size of approximately one cubic inch (~15cc) would be preferred. For samples of thin-coating materials such as paints, mastics, spray plasters, tapes, etc., a smaller sample size may be suitable for analysis. Generally, samples of insufficient volume should be **rejected**, and further analysis curtailed until the client is contacted. The quantity of sample affects the sensitivity of the analysis and reliability of the quantitation steps. If there is a question whether the sample is representative due to inhomogeneity, the sample should be rejected, at least until contacting the client to see if: 1) the client can provide more material or 2) the client wishes the laboratory to go ahead with the analysis, but with the laboratory including a statement on the limited sensitivity and reliability of quantitation. If the latter is the case, the report of analysis should state that the client was contacted, that the client decided that the lab should use less material than recommended by the method, and that the client acknowledges that this may have limited the sensitivity and quantitation of the method. At the time the client is contacted about the material, he or she should be informed that a statement reflecting these facts will be placed in the report.

2.1.1 Applicability

Stereomicroscopic analysis is applicable to all samples, although its use with vinyl floor tile, asphaltic products, etc., may be limited because of small asbestos fiber size and/or the presence of interfering components. It does not provide positive identification of asbestos.

2.1.2 Range

Asbestos may be detected at concentrations less than one percent by volume, but this detection is highly material dependent.

2.1.3 Interferences

Detection of possible asbestos fibers may be made more difficult by the presence of other nonasbestos fibrous components such as cellulose, fiber glass, etc., by binder/matrix materials which may mask or obscure fibrous components, and/or by exposure to conditions (acid environment, high temperature, etc.) capable of altering or transforming asbestos.

2.1.4 Precision and Accuracy

The precision and accuracy of these estimations are material dependent and must be determined by the individual laboratory for the percent range involved. These values may be determined for an individual analyst by the in-house preparation and analysis of standards and the use of error bars, control charts, etc.

The labs should also compare to National Voluntary Laboratory Accreditation Program (NVLAP) proficiency testing samples, if the lab participates in the Bulk Asbestos NVLAP, or to external quality assurance system consensus results such as from proficiency testing programs using characterized materials. However, at this time, consensus values for the quantity of asbestos have been shown to be unreliable. Only proficiency testing materials characterized by multiple techniques should be used to determine accuracy and precision.

2.1.5 Procedures

NOTE: Exposure to airborne asbestos fibers is a health hazard. Bulk samples submitted for analysis are oftentimes friable and may release fibers during handling or matrix reduction steps. All sample handling and examination must be carried out in a HEPA-filtered hood, a class 1 biohazard hood or a glove box with continuous airflow (negative pressure). Handling of samples without these precautions may result in exposure of the analyst to and contamination of samples by airborne fibers.

2.1.5.1 Sample Preparation

No sample preparation should be undertaken before initial stereomicroscopic examination. Distinct changes in texture or color on a stereomicroscopic scale that might denote an uneven distribution of components should be noted. When a sample consists of two or more distinct layers or building materials, each should be treated as a separate sample, when possible. Thin coatings of paint, rust, mastic, etc., that cannot be separated from the sample without compromising the layer are an exception to this case and may be included with the layer to which they are attached. Drying (by heat lamp, warm plate, etc.) of wet or damp samples is recommended before further stereomicroscopic examination and is mandatory before PLM examination. **Drying must be done in a safety hood.**

For nonlayered materials that are heterogeneous, homogenization by some means (mill, blender, mortar and pestle) may provide a more even distribution of sample components. It may also facilitate disaggregation of clumps and removal of binder from fibers (rarely however, it may mask fibers that were originally discernable).

For materials such as cementitious products and floor tiles, breaking, pulverizing, or grinding may improve the likelihood of exposing fibrous components.

It may be appropriate to treat some materials by dissolution with hydrochloric acid to remove binder/matrix materials. Components such as calcite, gypsum, magnesite, etc., may be removed by this method. For materials found to possess a high organic content (cellulose, organic binders), ashing by means of a muffle furnace or plasma asher (for small, cellulosic samples), or dissolution by solvents may be used to remove interfering material. In either case, it is recommended that matrix removal be tracked gravimetrically.

Additional information concerning homogenization, ashing and acid dissolution may be found in Sections 2.2.5.1 and 2.3.

2.1.5.2 Analysis

Samples should be examined with a simple stereomicroscope by viewing multiple fields of view over the entire sample. The whole sample should be observed after placement in a suitable container (watchglass, weigh boat, etc.) substrate. Samples that are very large should be subsampled. The sample should be probed, by turning pieces over and breaking open large clumps. The purpose of the stereomicroscopic analysis is to determine homogeneity, texture, friability, color, and the extent of fibrous components of the sample. This information should then be used as a guide to the selection of further, more definitive qualitative and quantitative asbestos analysis methods. Homogeneity refers to whether each subsample made for other analytical techniques (e.g. the "pinch" mount used for the PLM analysis), is likely to be similar or dissimilar. Color can be used to help determine homogeneity, whether the sample has become wet (rust color), and to help identify or clarify sample labelling confusion between the building material sampler and the laboratory. Texture refers to size, shape and arrangement of sample components. Friability may be indicated by the ease with which the sample is disaggregated (see definitions in Appendix A) as received by the analyst. This does not necessarily represent the friability of the material as determined by the assessor at the collection site. The relative proportion of fibrous components to binder/matrix material may be determined by comparison to similar materials of known fibrous content. For materials composed of distinct layers or two or more distinct building materials, each layer or distinct building material should be treated as a discrete sample. The relative proportion of each in the sample should be recorded. The layers or materials should then be separated and analyzed individually. Analysis results for each layer or distinct building material should be reported. If monitoring requirements call for one reported value, the results for the individual layers or materials should always be reported along with the combined value. Each layer or material should be checked for homogeneity during the stereomicroscopic analysis to determine the extent of sample preparation and homogenization necessary for successful PLM or other analysis. Fibers and other components should be removed for further qualitative PLM examination.

Using the information from the stereomicroscopic examination, selection of additional preparation and analytical procedures should be made. Stereomicroscopic examination should typically be performed again after any change or major preparation (ashing, acid dissolution, milling, etc.) to the sample. Stereomicroscopic examination for estimation of asbestos content may also be performed again after the qualitative techniques have clarified the identities of the various fibrous components to assist in resolving differences between the initial quantitative estimates made during the stereomicroscopic analysis and those of subsequent techniques. Calibration of analysts by use of materials of known asbestos content is essential.

The stereomicroscopic examination is often an iterative process. Initial examination and estimates of asbestos concentration should be made. The sample should then be analyzed by PLM and possibly other techniques. These results should be compared to the initial stereomicroscopic results. Where necessary, disagreements between results of the techniques should be resolved by reanalyzing the sample stereomicroscopically.

2.1.6 Calibration Materials

Calibration materials fall into several categories, including internal laboratory standards and other materials that have known asbestos weight percent content. These calibration materials could include:

- Actual bulk samples: asbestos-containing materials that have been characterized by other analytical methods such as XRD, AEM and/or gravimetry. (e.g. NVLAP test samples).

- Generated samples: in-house standards that can be prepared by mixing known quantities of asbestos and known quantities of asbestos-free matrix materials (by weight), and mixing (using blender, mill, etc.) thoroughly to achieve homogeneity; matrix materials such as vermiculite, perlite, sand, fiberglass, calcium carbonate, etc. may be used. A range of asbestos concentrations should be prepared (e.g. 1, 3, 5, 10, 20%, etc.). The relationship between specific gravities of the components used in standards should be considered so that weight/volume relationships may be determined.

- Photographs, drawings: photomicrographs of standards, computer-generated drawings, etc.

Suggested techniques for the preparation and use of in-house calibration standards are presented in Appendix C, and at greater length by Harvey et al.[1] The use of synthesized standards for analyst calibration and internal laboratory quality control is not new however, having been outlined by Webber et al.[2] in 1982.

2.1.7 References

1. Harvey, B. W., R. L. Perkins, J. G. Nickerson, A. J. Newland and M. E. Beard, "Formulating Bulk Asbestos Standards", **Asbestos Issues**, April 1991, pp. 22-29.

2. Webber, J. S., A. Pupons and J. M. Fleser, "Quality-Control Testing for Asbestos Analysis with Synthetic Bulk Materials". **American Industrial Hygiene Associations Journal**, 43, 1982, pp. 427-431.

2.2 Polarized Light Microscopy

2.2.1 Principle and Applicability

Samples of bulk building materials taken for asbestos identification should first be examined with the simple stereomicroscope to determine homogeneity and preliminary fiber identification. Subsamples should then be examined using PLM to determine optical properties of constituents and to provide positive identification of suspect fibers.

The principles of optical mineralogy are well-established.[1,2,3,4] A light microscope equipped with two polarizing filters is used to observe specific optical characteristics of a sample. The use of plane polarized light allows for the determination of refractive indices relative to specific crystallographic orientations. Morphology and color are also observed while viewing under plane polarized light. Observation of particles or fibers while oriented between polarizing filters whose privileged vibration directions are perpendicular (crossed polars) allows for determination of isotropism/anisotropism, extinction characteristics of anisotropic particles, and calculation of birefringence. A retardation plate may be placed in the polarized light path for verification of the sign of elongation. If subsamples are prepared in such a way as to represent all sample components and not just suspect fibers, semi-quantitative analysis may also be performed. Semi-quantitative analysis involves the use of calibrated visual area estimation and/or point counting. Visual area estimation is a semi-quantitative method that <u>must relate back to calibration materials</u>. Point counting, also semi-quantitative, is a standard technique used in petrography for determining the relative areas occupied by separate minerals in thin sections of rock. Background information on the use of point counting[3] and the interpretation of point count data[5] is available.

Although PLM analysis is the primary technique used for asbestos determination, it can show significant bias leading to false negatives and false positives for certain types of materials. PLM is limited by the visibility of the asbestos fibers. In some samples the fibers may be reduced to a diameter so small or masked by coatings to such an extent that they cannot be reliably observed or identified using PLM.

2.2.2 Range

The detection limit for visual estimation is a function of the quantity of sample analyzed, the nature of matrix interference, sample preparation, and fiber size and distribution. Asbestos may be detected in concentrations of less than one percent by area if sufficient material is analyzed. Since floor tiles may contain fibers too small to be resolved by PLM (< 0.25 μm in diameter), detection of those fibers by this method may not be possible. When point counting is used, the detection limit is directly proportional to the amount of sample analyzed, but is also limited by fiber visibility. Quantitation by area estimation, both visual and by point counting, should yield similar results if based on calibration standards.

2.2.3 Interferences

Fibrous and nonfibrous, organic and inorganic constituents of bulk samples may interfere with the identification and quantitation of the asbestos mineral content. Binder/matrix materials may coat fibers, affect color, or obscure optical characteristics to the extent of masking fiber identity. Many organic mastics are soluble in refractive index liquids and, unless removed prior to PLM examination, may affect the refractive index measurement of constituent materials. Fine particles of other materials may also adhere to fibers to an extent sufficient to cause confusion in identification. Gravimetric procedures for the removal of interfering materials are presented in Section 2.3.

2.2.4 Precision and Accuracy

Data obtained for samples containing a single asbestos type in a sample matrix have been reported previously by Brantley et al.[6] Data for establishing the accuracy and precision of the method for samples with various matrices have recently become available. Perkins,[7] Webber et al.[8] and Harvey et al.[9] have each documented the tendency for visual estimates to be high when compared to point-count data. Precision and accuracy must be determined by the individual laboratory for the percent range involved. If point counting and/or visual estimates are used, a table of reasonably expanded errors, such as those shown in Table 2-1, should be generated for different concentrations of asbestos.

If the laboratory cannot demonstrate adequate precision and accuracy (documented by control charts, etc), quantitation by additional methods, such as gravimetry, may be required. Refer to the Handbook for SRM Users[10] for additional information concerning the concepts of precision and accuracy.

TABLE 2-1. SUGGESTED ACCEPTABLE ERRORS FOR PLM ANALYSIS
(Based on 400 point counts of a reasonably homogeneous sample or 100 fields of view for visual estimate)

% Area Asbestos	Acceptable Mean Result	% Area Asbestos	Acceptable Mean Result
1	>0-3%	50	40-60%
5	>1-9%	60	50-70%
10	5-15%	70	60-80%
20	10-30%	80	70-90%
30	20-40%	90	80-100%
40	30-50%	100	90-100%

2.2.5 Procedures

NOTE: Exposure to airborne asbestos fibers is a health hazard. Bulk samples submitted for analysis are oftentimes friable and may release fibers during handling or matrix reduction steps. All sample and slide preparations must be carried out in a HEPA-filtered, a class 1 biohazard hood, or a glove box with continuous airflow (negative pressure). Handling of samples without these precautions may result in exposure of the analyst to and contamination of samples by airborne fibers.

2.2.5.1 Sample Preparation

Slide mounts are prepared for the identification and quantitation of asbestos in the sample.

2.2.5.1.1 Qualitative Analysis Preparation

The qualitative preparation must allow the PLM analysis to classify the fibrous components of the sample as asbestos or nonasbestos. The major goal of the qualitative preparation is to mount easily visible fibers in appropriate refractive index liquids for complete optical characterization. Often this can be accomplished by making immersion grain mounts of random subsamples of the homogeneous material. Immersion liquids with refractive indices close to the suspected (see stereomicroscopic analysis) asbestos mineral should be used for the qualitative analysis so that n_D can be determined. Problem samples include those with inhomogeneities, coatings, small fibers, and interfering compounds. Additional qualitative preparations are often necessary for these types of samples. All samples, but especially those lacking homogeneity, may require picking of fibers from specific sample areas during the stereomicroscopic examination. Coatings on the fibers often need to be removed by mechanical or chemical means. Teasing the particles apart or use of a mortar and pestle or similar mechanical method often is sufficient to free fibers from coatings. Chemical means of removing some coatings and interfering compounds are discussed in Section 2.3, Gravimetry.

2.2.5.1.2 Quantitative Analysis Preparation

The major purpose of the quantitative preparation is to provide the analyst with a representative grain mount of the sample in which the asbestos can be observed and distinguished from the nonasbestos matrix. This is typically performed by using randomly selected subsamples from a homogeneous sample (see stereomicroscopic analysis). Particles should be mounted in a refractive index (RI) liquid that allows the asbestos to be visible and distinguished from nonasbestos components. Care should be taken to ensure proper loading and even distribution of particles. Both the qualitative and quantitative sample preparations are often iterative processes. Initial samples are prepared and analyzed. The PLM analysis may disclose problems or raise questions that can only be resolved by further preparations (e.g. through the use of different RI immersion liquids, elimination of interfering compounds, sample homogenization, etc.)

For layered materials, subsamples should be taken from each individual or discrete layer. Each of these subsamples should be treated as a discrete sample, but as stated in Section 2.1.5.2, the results for the individual layers or materials may be combined if called for by monitoring requirements.

Homogenization involves the use of any of a variety of devices, such as a mortar and pestle, mill, or blender to pulverize, disaggregate and mix heterogeneous, friable bulk materials. Selection of the appropriate device is dependent upon personal preference and the nature of the materials encountered. A blender or mortar and pestle may be adequate for homogenizing materials that lack appreciable amounts of tacky matrix/binder, and for separating interfering components from the fibers. For materials which are unusually sticky or tacky, or contain unusually long asbestos fibers, milling (especially freezer milling) may be more efficient. However, milling should be discontinued as soon as the material being milled appears homogeneous, in order to reduce the potential for mechanically reducing fiber size below the resolving power of the polarizing microscope. Hammer mills or cutting mills may also be used on these materials; however, the same precaution regarding reduction of fiber size should be taken. Blending /milling devices should be disassembled (to the extent possible) and thoroughly cleaned after each use to minimize contamination.

2.2.5.2 Analysis

Analysis of bulk building materials consists of the identification and semi-quantitation of the asbestos type(s) present, along with the identification, where possible, of fibrous nonasbestos materials, mineral components and matrix materials. If the sample is heterogeneous due to the presence of discrete layers or two or more distinct building materials, each layer or distinct material should be analyzed, and results reported. Total asbestos content may also be stated in terms of a relative percentage of the total sample.

2.2.5.2.1 Identification

Positive identification of asbestos requires the determination of the following optical properties:

- Morphology
- Color and, if present, pleochroism
- Refractive indices ($\pm$ .005)
- Birefringence
- Extinction characteristics
- Sign of elongation

Descriptions of the optical properties listed above for asbestos fibers may be found in Appendix A, Glossary of Terms. Table 2-2 lists the above properties for the six types of asbestos and Table 2-3 presents the central stop dispersion staining colors for the asbestos minerals with selected high-dispersion index liquids. Tables 2-4 and 2-5 list selected optical properties of several mineral and man-made fibers. All fibrous materials in amounts greater than trace should be identified as asbestos or nonasbestos, with all optical properties measured for asbestos and at least one optical property measured for each nonasbestos fibrous component that will distinguish each from asbestos. Small fiber size and/or binder may necessitate viewing the sample at higher magnification (400-500x) than routinely used (100x).

Although it is not the purpose of this section to explain the principles of optical mineralogy, some discussion of the determination of refractive indices is warranted due to its importance to the proper identification of the asbestos minerals. Following is a brief discussion of refractive index determination for the asbestos minerals.

All asbestos minerals are anisotropic, meaning that they exhibit different optical properties (including indices of refraction) in different directions. All asbestos minerals are biaxial, meaning that they have one principal refractive index parallel (or nearly parallel) to the length of the fiber and two principal refractive indices (plus all intermediate indices between these two) in the plane perpendicular (or nearly so) to the length of the fiber. Although chrysotile (serpentine) is classified as a biaxial mineral, it behaves as a uniaxial mineral (two principal refractive indices) due to its scrolled structure. Amosite and crocidolite, although also biaxial, exhibit uniaxial properties due to twinning of the crystal structure and/or random orientation of fibrils in a bundle around the long axis of the bundle. For all of the asbestos minerals except crocidolite, the highest refractive index (γ) is aligned with the fiber length (positive sign of elongation). For crocidolite, the lowest refractive index (α) is aligned with the fiber length (negative sign of elongation). A more complete explanation of the relationship of refractive indices to the crystallographic directions of the asbestos minerals may be found in References 1, 2, 4, 11 and 12. It should be noted that for the measurement of refractive indices in an anisotropic particle (e.g. asbestos fibers), the orientation of the particle is quite critical. Orientation with respect to rotation about the axis of the microscope (and thus with respect to the vibration directions of the polarizer and analyzer) and also to the horizontal plane (plane of the microscope stage) will affect the determination of the correct values for refractive indices. The refractive index that is measured will always correspond to a direction perpendicular to the axis of the microscope (i.e., lying in the plane of the stage) and is the direction in that horizontal plane parallel to the vibration direction of the polarizer, by convention E-W.

To determine $\gamma(n \parallel)$ for chrysotile, anthophyllite and amosite, the index is measured when the length of the fiber is aligned parallel to the vibration direction of the polarizer (E-W). Under crossed polars, the fiber should be at extinction in this orientation. To determine the lowest refractive index, α $(n \perp)$, for chrysotile and amosite, the fiber should be oriented N-S (extinction position under crossed polars). The determination of $n \parallel$ and $n \perp$ with crocidolite is accomplished in the same manner as with amosite and chrysotile with the exception that the α and γ directions are reversed. For crocidolite, α is measured at the E-W position (parallel to the polarizer) and γ is measured at the N-S orientation (perpendicular to the polarizer). For anthophyllite, the fiber should be oriented N-S and the lowest and highest indices for this orientation should be measured. These correspond to α and β respectively.

The extinction behavior of tremolite-actinolite is anomalous compared to that of most monoclinic minerals due to the orientation of the optic axes relative to the crystallographic axes. This relationship is such that the refractive indices of the principal axes α and γ <u>are not measured</u> when the fiber is exhibiting the maximum extinction angle. The values measured at these positions are α' and γ'. The fiber exhibits an extinction angle within a few degrees of the maximum throughout most of its rotation. A wide range of refractive indices from α' to α, and from γ' to γ, are observed. For tremolite-actinolite, β is measured on those fibers displaying parallel extinction when oriented in the N-S position. The refractive index for α is also measured when the fiber is oriented generally in the N-S position and exhibits <u>the true extinction</u> angle; true α will be the <u>minimum</u> index. To determine the refractive index for γ, the fibers should be oriented E-W and exhibit the true extinction angle; true γ will be the <u>maximum</u> value for this orientation.

When viewing single fibers, the analyst may often be able to manipulate the microscope slide cover slip and "roll" the fibers to positions that facilitate measuring the true values of refractive indices. When viewing a large population of fibers with the microscope in the dispersion staining mode, the analyst can easily detect fibers that exhibit the highest and lowest indices (β and α) in the N-S position and the highest indices (γ) in the E-W position. Since individual asbestos fibrils cannot generally be resolved using polarized light microscopy, refractive indices are most commonly measured on fiber bundles. Such measurements would not result in true values for the indices and therefore by convention should be reported as α' and γ'.

Asbestos types chrysotile, amosite and crocidolite are currently available as SRM 1866 and actinolite, tremolite and anthophyllite as SRM 1867 from the Office of Standard Reference Materials, National Institute of Standards and Technology.

2.2.5.2.2 Quantitation of Asbestos Content

As described in Sections 2.1.5 and 2.1.6, a calibrated visual volume estimation of the relative concentrations of asbestos and nonasbestos components should be made during the stereomicroscopic examination. In addition, quantitation of asbestos content should be performed on subsample slide mounts using calibrated visual area estimates and/or a point counting procedure. Section 2.1.6 and Appendix C discuss the procedures for preparation and use of calibration standards. After thorough PLM analysis in which the asbestos and other components of the bulk material are identified, several slides should be carefully prepared from randomly selected subsamples. If the sample is not homogeneous, some homogenization procedure should be performed to ensure that slide preparations made from small pinch samples are representative of the total sample. Homogenization may range from gentle mixing using a mortar and pestle to a brief period of mixing using a blender equipped with a mini-sample container. The homogenization should be of short duration (~ 15 seconds) if using the blender technique so as to preclude a significant reduction in fiber size. The use of large cover slips (22x30mm) allows for large subsamples to be analyzed. Each slide should be checked to ensure that the subsample is representative, uniformly dispersed, and loaded in a way so as not to be dominated by superimposed (overlapping) particles.

During the qualitative analysis of the sample, the analyst should decide on the appropriate optical system (including magnification) to maximize the visibility of the asbestos in the sample while still allowing the asbestos to be uniquely distinguished from the matrix materials. The analyst may choose to alter the mounting medium or the optical system to enhance contrast. During the quantitative analysis, slides should be scanned using an optical setup that yields the best visibility of the asbestos. Upon finding asbestos, the parameters that were selected in the qualitative analysis for uniquely distinguishing it from the matrix should be used for identification. These properties will vary with the sample but include any or all of the parameters required for the qualitative analysis. For instance, low magnification allows for concurrent use of dispersion staining (focal screening), but compromises resolution of extremely small diameter fibers; use of a compensator plate and crossed polarizers frequently enhances the contrast between asbestos fibers and matrix material.

Visual area estimates should be made by comparison of the sample to calibration materials that have similar textures and fiber abundance (see Section 2.1.6 and Appendix C). A minimum of three slide mounts should be examined to determine the asbestos content by visual area estimation. Each slide should be scanned in its entirety and the relative proportions of asbestos and nonasbestos noted. It is suggested that the ratio of asbestos to nonasbestos material be recorded for several fields for each slide and the results be compared to data derived from the analysis of calibration materials having similar textures and asbestos content.

For point counting, an ocular reticle (cross-line or point array) should be used to visually superimpose a point or points on the microscope field of view. The cross-line reticle is preferred. Its use requires the scanning of most, if not all, of the slide area, thereby minimizing bias that might result from lack of homogeneity in the slide preparation. In conjunction with this reticle, a click-stop counting stage can be used to preclude introducing bias during slide advancement. Magnification used will be dictated by fiber visibility. The slide should be examined along multiple parallel traverses that adequately cover the sample area. The analyst should score (count) only points directly over occupied (nonempty) areas. Empty points should not be scored on the basis of the closest particle. If an asbestos fiber and a nonasbestos particle overlap so that a point is superimposed on their visual intersection, a point should be scored for both categories. If the point(s) is/are superimposed on an area which has several overlapping particles, the slide should be moved to another field. While not including them in the total asbestos points counted, the analyst should record the presence of any asbestos detected but not lying under the reticle cross-line or array points. A minimum of 400 counts (maximum of eight slides with 50 counts each to minimum of two slides with 200 counts each) per sample is suggested, but it should be noted that accuracy and precision improve with number of counts. Point counting provides a determination of the projected area percent asbestos. Conversion of area percent to dry weight percent is not feasible unless the specific gravities and relative volumes of the different materials are known. It should be noted that the total amount of material to be analyzed is dependent on the asbestos concentration, i.e. the lower the concentration of asbestos, the larger the amount of sample that should be analyzed, in both the visual estimation and point counting methods. Quantitation by either method is made more difficult by low asbestos concentration, small fiber size, and presence of interfering materials.

It is suggested that asbestos concentration be reported as volume percent, weight percent or area percent depending on the method of quantitation used. A weight concentration cannot be determined without knowing the relative specific gravities and volumes of the sample components.

TABLE 2-2. OPTICAL PROPERTIES OF ASBESTOS FIBERS

Mineral	Morphology and Color[1]	Refractive Indices[2] α	Refractive Indices[2] γ[5]	Birefringence[6]	Extinction	Sign of Elongation
Chrysotile (asbestiform serpentine)	Wavy fibers. Fiber bundles have splayed ends and "kinks". Aspect ratio typically >10:1. Colorless[3]	1.493-1.546 1.532-1.549 1.529-1.559 1.544-1.553	1.517-1.557 1.545-1.556 1.537-1.567 1.552-1.561	0.004-0.017	Parallel	+ (length slow)
Amosite (asbestiform grunerite)	Straight to curved, rigid fibers. Aspect ratio typically >10:1. Colorless to brown, nonpleochroic or weakly so.[4] Opaque inclusions may be present	1.657-1.663 1.663-1.686 1.663-1.686 1.676-1.683	1.699-1.717 1.696-1.729 1.696-1.729 1.697-1.704	0.021-0.054	Usually parallel	+ (length slow)
Crocidolite (asbestiform riebeckite)	Straight to curved, rigid fibers. Aspect ratio typically > 10:1. Thick fibers and bundles common, blue to dark-blue in color. Pleochroic.	1.693 1.654-1.701 1.680-1.698	1.697 1.668-1.717 1.685-1.706	0.003-0.022	Usually parallel	- (length fast)
Anthophyllite-asbestos	Straight to curved fibers and bundles. Aspect ratio typically > 10:1. Anthophyllite cleavage fragments may be present with aspect ratios <10:1. Colorless to light brown.	1.598-1.652 1.596-1.694 1.598-1.674 1.6148[7]	1.623-1.676 1.615-1.722 1.615-1.697 1.6362[7]	0.013-0.028	Parallel	+ (length slow)
Tremolite-Actinolite-asbestos	Straight to curved fibers and bundles. Aspect ratio typically > 10:1. Cleavage fragments may be present with aspect ratios <10:1. Colorless to pale green	Tremolite 1.600-1.628 1.604-1.612 1.599-1.612 1.6063[7] Actinolite 1.600-1.628 1.612-1.668 1.613-1.628 1.6126[7]	 1.625-1.655 1.627-1.635 1.625-1.637 1.6343[7] 1.625-1.655 1.635-1.688 1.638-1.655 1.6393[7]	0.017-0.028 (Tremolite) 0.017-0.028 (Actinolite)	Parallel and oblique (up to 21°); Composite fibers show parallel extinction.	+ (length slow)

[1]Colors cited are seen by observation with plane polarized light.

[2]From references 2, 11, 12, and 18, respectively. Refractive indices for n_d at 589.3nm.

[3]Fibers subjected to heating may be brownish. (references 13, 14, and 15)

[4]Fibers subjected to heating may be dark brown and pleochroic. (references 13, 14, and 15)

[5]‖ to fiber length, except ⊥ to fiber length for crocidolite only.

[6]Maximum and minimum values from references 2, 11, 12, and 18 given.

[7]± 0.0007

TABLE 2-3. TYPICAL CENTRAL STOP DISPERSION STAINING COLORS[1]

Mineral	Cargille* RI Liquid	n∥	n⊥
Chrysotile	1.550HD	Magenta to light blue-green λ_0's ca. 520-620nm	Blue-green to pale blue λ_0's ca. 600-700nm
Amosite	1.680	Yellow to magenta λ_0's ca. 420-520nm	Blue magenta to light blue λ_0's ca. 560-660nm
Crocidolite	1.680	Yellow to magenta λ_0's ca. 420-520nm	Pale yellow to golden yellow λ_0's ca. 360-460nm
Anthophyllite-asbestos	1.605HD	Pale yellow to yellow λ_0's ca. 330-430nm	Golden yellow to light blue green λ_0's ca. 460-700nm
Tremolite-asbestos	1.605HD	Pale yellow to yellow λ_0's ca. 330-430nm	Golden yellow to light blue green λ_0's ca. 460-700nm
Actinolite-asbestos	1.605HD	Pale yellow λ_0's ca. 260-360nm	Pale yellow to golden yellow λ_0's ca. 360-460nm
	1.630HD	Yellow to magenta λ_0's ca. 420-520nm	Golden yellow to blue λ_0's ca. 450-600nm

[1]Modified from reference 16

TABLE 2-4. OPTICAL PROPERTIES OF MAN-MADE TEXTILE FIBERS[1,2]

Fiber Type	n∥	n⊥	n∥ - n⊥	Sign of Elongation
Polyester (Dacron®)	1.710	1.535	0.175	+
Polyamide (Nylon®)	1.582	1.514	0.063	+
Aramid (Kevlar®)	≈2.37	≈1.641	0.729	+
Olefin (Polyethylene)	1.556	1.512	0.044	+
Olefin (Polypropylene)	1.520	1.495	0.025	+
Viscose Rayon	1.535-1.555	1.515-1.535	0.020	+
Acetate	1.478-1.480	1.473-1.476	0.004-0.005	+
Acrylic (Orlon®)	1.505-1.515	1.507-1.517	0.004-0.002	-
Modacrylic (Dynel®)	1.535	1.532	0.002	+

[1]Modified from reference 17

[2]Refractive indices for specific fibers; other fibers may vary

TABLE 2-5. OPTICAL PROPERTIES OF SELECTED FIBERS[1]

FIBER TYPE	MORPHOLOGY	REFRACTIVE INDICES	BIREFRINGENCE ($n\parallel - n\perp$)	EXTINCTION ANGLE	SIGN OF ELONGATION	DISPERSION STAINING COLORS
Paper (Cellulose)	Tapered, flat ribbons	$n\parallel \sim 1.580$ $n\perp \sim 1.530$	High (0.05)	Parallel and incomplete	+	in 1.550HD $n\parallel$: yellow (λ_0's < 400nm) $n\perp$: pale blue (λ_0's > 700nm)
Olefin (polyethylene)	Filaments or shredded like chrysotile	$n\parallel \sim 1.556$ $n\perp \sim 1.512$	Moderate (0.044)	Parallel	+	in 1.550HD $n\parallel$: yellow to magenta (λ_0's = 440-540nm) $n\perp$: pale blue (λ_0's > 700nm)
Brucite (nemalite)	Straight fibers	$n\parallel \sim 1.560$-1.590 $n\perp \sim 1.580$-1.600	Moderate (0.012-0.020)	Usually parallel	- occasionally +	in 1.550HD $n\parallel$: golden yellow (λ_0's 440-460nm) $n\perp$: yellow (λ_0's 400-440nm)
Heated amosite	Similar to unheated, (brittle and shorter) pleochroic: $n\parallel$-dark brown $n\perp$ yellow	$n\parallel$ and $n\perp$ > 1.700[2]	High (> 0.05)	Usually parallel	+	in 1.680HD $n\parallel$ & $n\perp$: both pale yellow to white (λ_0's < 400nm)
Glass fibers, Mineral wool	Exotic shapes, tear drops, single filaments	1.515-1.700	Isotropic	------	-	in 1.550HD usually pale blue to blue (λ_0's 580 to > 700nm)
Wollastonite	Straight needles and blades	$n\parallel \sim 1.630$ $n\perp \sim 1.632$ $n\perp$ also 1.610	Moderate to low (0.018 to 0.002)	Parallel and oblique	+ and -	in 1.605HD $n\parallel$ & $n\perp$: yellow to pale yellow (λ_0's < 460nm)
Fibrous talc	Thin cleavage ribbons and wavy fibers	$n\parallel \sim 1.60$ $n\perp \sim 1.54$	High (0.06)	Parallel and oblique	+	in 1.550HD $n\parallel$: pale yellow (λ_0's < 400nm) $n\perp$: pale blue (λ_0's > 660nm)

[1]From reference 19

[2]From references 13, 14, and 15

2.2.5.2.3 Microscope Alignment

In order to accurately measure the required optical properties, a properly aligned polarized light microscope must be utilized. The microscope is aligned when:

1) the privileged directions of the substage polarizer and the analyzer are at 90° to one another and are represented by the ocular cross-lines;

2) the compensator plate's privileged vibration directions are 45° to the privileged directions of the polarizer and analyzer;

3) the objectives are centered with respect to stage rotation; and,

4) the substage condenser and iris diaphragm are centered in the optic axis.

Additionally, the accurate measurement of the refractive index of a substance requires the use of calibrated refractive index liquids. These liquids should be calibrated regularly to an accuracy of 0.004, with a temperature accuracy of 2°C using a refractometer or R.I. glass beads.

2.2.6 References

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2.3 Gravimetry

2.3.1 Principle and Applicability

Many components of bulk building materials, specifically binder components, can be selectively removed using appropriate solvents or, in the case of some organics, by ashing. The removal of these components serves the following purposes:

1) to isolate asbestos from the sample, allowing its weight to be determined;

2) to concentrate asbestos and therefore lower the detection limit in the total sample;

3) to aid in the detection and identification of fibrous components; and,

4) to remove organic (ashable) fibers which are optically similar to asbestos.

Common binder materials which are removed easily using the techniques described include: 1) calcite, gypsum, magnesite, brucite, bassanite, portlandite, and dolomite, using hydrochloric acid, and 2) vinyl, cellulose, and other organic components, by ashing. The removal of the binder components results in a residue containing asbestos, if initially present, and any other non-soluble or non-ashable components which were present in the original sample. Unless the procedures employed result in the loss of some asbestos, the weight percent of the residue is the upper limit for the weight percent of asbestos in the sample.

This section describes the procedure for removing acid-soluble and ashable components, and for determining the weight percent of the residue. However, the acid dissolution and ashing techniques can be used without the accompanying weight measurements to either liberate or clean fibers to aid in qualitative PLM or AEM analyses.

This technique is not an identification technique. Other methods, such as PLM, XRD, or AEM must be used to determine the identity of the components. A description of the suggested apparatus, reagents, etc. needed for the techniques described is included in Appendix B.

2.3.2 Interferences

Any components which cannot by removed from the sample by selective dissolution or ashing interfere with asbestos quantitation. These components include, but are not limited to, many silicates (micas, glass fibers, etc.) and oxides ($Ti0_2$, magnetite, etc.). When interfering phases are present (the residue contains other phases in addition to asbestos), other techniques such as PLM, AEM, or XRD must be used to determine the percent of asbestos in the residue.

Care must be taken to prevent loss of or chemical/structural changes in the critical components (asbestos). Prolonged exposure to acids or excessive heating (above 500°C) can cause changes in the asbestos components in the sample and affect the optical properties.[1,2,3]

2.3.3 Quantitation

The weight of the residue remaining after solvent dissolution/ashing should be compared with the original weight of the material. Presuming no insoluble material is lost, the weight percent of the residue is the upper limit for the amount of asbestos in the sample. If the residue is comprised only of asbestos, then the weight percent of residue equals the weight percent of asbestos in the sample. If the residue contains other phases, then techniques such as PLM, XRD, or AEM must be employed to determine the relative abundance of asbestos in the residue.

The precision and accuracy of the technique are dependent upon the homogeneity of the material, the accuracy of the weight measurements, and the effectiveness of the sample reduction and filtering procedures. In practice, the precision can be equal to ±1%, and the accuracy at 1 wt% asbestos can be less than or equal to ±10% relative.

The incomplete solution of components and the presence of other nonasbestos components in the residue contribute to producing a positive bias for the technique (falsely high percentages of asbestos).

2.3.4 Preliminary Examination and Evaluation

Stereomicroscopic and PLM examinations of the sample should already have been conducted prior to initiating this procedure. These examinations should have provided information about: 1) whether the sample contains components which can be removed by acid-washing, solvent dissolution, or ashing, and 2) whether the sample contains asbestos, or fibers that might be asbestos, or whether no asbestos was detected.

If the sample is friable and contains organic (ashable) components, the ashing procedure should be followed. If the sample is friable and contains HCl-soluble components, the acid dissolution procedure should be followed. If the sample is friable and contains both types of components, the two procedures can be applied, preferably with acid dissolution following ashing.

If the sample is nonfriable (e.g. floor tiles), it is also recommended that the ashing procedure be used first, followed by the acid dissolution procedure. The ashing procedure reduces floor tiles to a material which is easily powdered, simplifying the sample preparation for acid dissolution.

2.3.5 Sample Preparation

2.3.5.1 Drying

Any moisture in the sample will affect the weight measurements, producing falsely low percentages of residue. If the sample is obviously wet, it should be dried at low temperature (using a heat lamp, or simply by exposure at ambient conditions, prior to starting the weighing procedure). If an oven is used, the drying temperature should not exceed 60°C. Drying by means of heat lamp or ambient air must be performed within a safety-filtered hood. Even if the sample appears dry, it can contain enough moisture to affect the precision and accuracy of the technique. The test for sample moisture involves placing the amount of sample to be used on the weighing pan; if the weight remains stable with time, then the sample is dry enough. If the weight decreases as the sample sits on the weighing pan, then the sample should be dried. Where conditions of moderate to high humidity are known to exist, all materials to be weighed should be allowed time to stabilize to these ambient conditions.

2.3.5.2 Homogenization/Grain Size Reduction

To increase the accuracy and precision of the acid dissolution technique, the sample should be homogenized prior to analysis. This reduces the grain size of the binder material and releases it from fiber bundles so that it may be dissolved in a shorter time period. Leaving the sample in the acid for a longer period of time to complete the dissolution process can adversely affect the asbestos components, and is not recommended. Homogenization of the sample also ensures that any material removed for analysis will more likely be representative of the entire sample.

Homogenization of friable samples prior to ashing may also accelerate the ashing process; however, the ashing time can simply be increased without affecting the asbestos in the sample. Nonfriable samples, such as vinyl floor tiles, can be broken or shaved into pieces to increase surface area and accelerate the ashing process.

Homogenization and grain size reduction can be accomplished in a variety of ways: 1) hand grinding in a mortar and pestle; 2) crushing with pliers or similar instrument; 3) mixing in a blender; 4) milling (i.e. Wylie mill, cryomill, etc.); or 5) any other technique which seems suitable. If the fibers are extremely long, a pair of scissors or similar implement can be used to reduce the fiber length.

2.3.6 Procedure for Ashing

1) **Weigh appropriate amount of material.**

   There is no restriction on the maximum weight of material used; however, a large amount of material may take longer to ash. Enough material should be used to avoid a significant contribution of weighing errors to the total accuracy and precision.

2) **Place material in crucible, weigh, and cover with lid.**

   Placing a lid on the crucible both minimizes the amount of oxygen available, slowing the rate of combustion of the sample, and prevents any foreign material from falling into the crucible during ashing.

3) **Place crucible into furnace, and ash for at least 6 hours.**

   The furnace temperature at the sample position should be at least 300°C but should not exceed 500°C. If the sample combusts (burns), the temperature of the sample may exceed 500°C. Chrysotile will decompose above approximately 500°C.

   The furnace area should be well-ventilated and the fumes produced by ashing should be exhausted outside the building.

   The ashing time is dependent on the furnace temperature, the amount of sample, and the surface area (grain size). Six hours at 450°C is usually sufficient.

4) **Remove crucible from furnace, allow contents to adjust to room temperature and humidity, and weigh.**

5) **Divide residue weight by starting weight and multiply by 100 to determine weight% residue.**

6) **Analyze residue and/or proceed to acid dissolution procedure.**

If the objective was to remove organic fibers that may be confused optically with asbestos, examine residue with PLM to determine whether any fibers remain.

If the sample is a floor tile, the acid dissolution procedure must now be performed. The residue does not have to be analyzed at this stage.

2.3.7 Use of Solvents for Removal of Organics

Solvent dissolution may be used as a substitute for low temperature ashing for the purpose of removing organic interferences from bulk building materials. However, solvent dissolution, because of the involvement of potentially hazardous reagents such as tetrahydrofuran, amyl acetate, 1-1-1, trichlorethane, etc., requires **that all work be performed with extreme caution inside a biohazard hood.** Material Safety Data Sheets should be reviewed before using any solvent. Solvent dissolution involves more apparatus than does ashing, and requires more time, mainly due to set-up and slow filtration resulting from viscous solvent/residue mixtures.

The following is a brief description of the solvent dissolution process.

1) **Weigh starting material.**

Place approximately 15-25ml of solvent in a 100ml beaker. Add 2.5-3.0 grams (carefully weighed for continued gravimetric tracking) of powdered sample.

2) **Untrasonicate sample.**

Place the beaker in an ultrasonic bath (or ultrasonic stirrer) for approximately 0.5 hours. The sample containers should be covered to preclude escape of an aerosol spray.

3) **Centrifuge sample.**

Weigh centrifuge vial before adding beaker ingredients. Wash beaker with an additional 10-15ml of solvent to remove any remaining concentrate. Then centrifuge at approximately 2000-2500 rpm for 0.5 hour. Use solvent-resistant centrifuge tubes.

4) **Decant sample, reweigh.**

After separation by centrifuging, decant solvent by pipetting. Leave a small amount of solvent in the centrifuge vial to minimize the risk of decanting solid concentrate. Allow solid concentrate to dry in vial, then reweigh.

2.3.8 Procedure for Acid Dissolution

1) **Weigh starting material, transfer to acid resistant container.**

Small, dry sample weights between 0.1g and 0.5g are recommended (determined for 47mm filters - adjust amount if different diameter filters are used). If too much material is left after acid dissolution the filter can get clogged and prevent complete filtration. Very small samples are also to be avoided, as the weighing errors will have a large effect on the total accuracy and precision of the technique.

2) **Weigh filter.**

3) **Add HCl to sample in container, stir, allow to sit for 2-10 minutes.**

Either concentrated or dilute HCl can be used. If concentrated HCl is used, add enough acid to completely soak the material, allow the reaction to proceed to completion, and then dilute with distilled water. Alternatively, a dilute solution, made by adding concentrated HCl to distilled water, can be used in the place of concentrated HCl. A solution of 1 part concentrated HCl to 3 parts distilled water (approximately 3N solution) has been found to be quite effective in removing components within 5 minutes. For a sample size less than 0.5g, 20-30 ml of a 3N HCl solution is appropriate. In either case (using concentrated or dilute HCl), the reaction will be more effective if the sample has been homogenized first. All obvious signs of reaction (bubbling) should cease before the sample is filtered. Add fresh acid, a ml or two at a time, to ensure complete reaction. It should be noted that if dolomite is present, a 15-20 minute exposure to concentrated HCl may be required to completely dissolve the carbonate materials.

NOTE: Other solvents may be useful for selective dissolution of nonasbestos components. For example, acetic acid will dissolve calcite, and will not dissolve asbestos minerals. If any solvent other than hydrochloric acid is used for the dissolution of inorganic components, the laboratory must be able to demonstrate that the solvent does not remove asbestos from the sample.

4) **Filter solution.**

Use the pre-weighed filter. Pour the solution into the vacuum filter assembly, then rinse all material from container into filter assembly. Rinse down the inside walls of the glass filter basin and check ιor particles clinging to the basin after removal.

5) **Weigh dried filter + residue, subtract weight of filter from total.**

6) **Divide residue weight by starting weight and multiply by 100 to determine weight% residue.**

7) **Analyze residue.**

Perform stereomicroscopic examination of residue (can be performed without removing the residue from the filter). Note in particular whether any binder material is still present.

Perform PLM, AEM, or XRD analysis of residue to identify fibers and determine concentration as described in the appropriate sections of this method.

8) **Modify procedure if necessary.**

If removal of the acid soluble components was not complete, start with a new subsample of material and try any of the following:

a) Decrease grain size of material (by grinding, milling, etc.)
b) Put solutions on hot plate - warm slightly
c) Increase soak time (exercise caution)

9) **Calculate relative weight% asbestos in sample.**

wt% asbestos in sample = % asbestos in residue x wt% residue ÷ 100

For floor tiles, if the ashing procedure was used first, multiply the weight % of asbestos in the sample, as determined above, by the weight percent of the residue from the ashing procedure, then divide by 100.

Example:
A = wt% residue from ashing = 70%
B = wt% residue from HCl = 20%
C = wt% of asbestos in HCl residue = 50%

wt% asbestos after HCl dissolution = B x C ÷ 100 = 20 x 50 ÷ 100 = 10%

wt% asbestos in floor tile = (B x C ÷ 100) x A ÷ 100 = 10 x 70 ÷ 100 = 7%

If weights are expressed in decimal form, multiply the weight % of asbestos in the sample by the weight % of the residue from the ashing procedure, then multiply by 100.

wt% asbestos after HCl dissolution = B x C = 0.2 x 0.5 = 0.1 (x 100 = 10%)

wt% asbestos in floor tile = (B x C) x A = 0.1 x 0.7 = 0.07 (x 100 = 7%)

2.3.9 Determination of Optimal Precision and Accuracy

The precision of the technique can be determined by extracting multiple subsamples from the original sample and applying the same procedure to each. The optimal accuracy of the technique can be determined by applying gravimetric standards. Mixtures of calcite and asbestos (chrysotile, amosite, etc.) in the following proportions are recommended for testing the accuracy of the acid dissolution technique: 0.1 wt% asbestos/99.9 wt% calcite, 1.0 wt% asbestos/99.0 wt% calcite, and 10 wt% asbestos/90 wt% calcite. Mixtures of cellulose and asbestos are useful for testing the accuracy of the ashing technique.

Mixtures of only two components, as described above, are simplifications of "real-world" samples. The accuracy determined by analyzing these mixtures is considered optimal and may not apply directly to the measurement of each unknown sample. However, analyzing replicates and standards using the full laboratory procedure, including homogenization, ashing, acid dissolution, filtration, and weighing, may uncover steps that introduce significant bias or variation that the laboratory may then correct.

2.3.10 References

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2.4 X-Ray Powder Diffraction

2.4.1 Principle and Applicability

The principle of x-ray powder diffraction (XRD) analysis is well established.[1,2] Any solid crystalline material will diffract an incident beam of parallel, monochromatic x-rays whenever Bragg's Law,

$$\lambda = 2d \sin \theta,$$

is satisfied for a particular set of planes in the crystal lattice, where

λ = the x-ray wavelength, Å;
d = the interplanar spacings of the set of reflecting lattice planes, Å and
θ = the angle of incidence between the x-ray beam and the reflecting lattice planes.

By appropriate orientation of a sample relative to the incident x-ray beam, a diffraction pattern can be generated that will be uniquely characteristic of the structure of the crystalline phases present.

Unlike optical methods of analysis, however, XRD cannot determine crystal morphology. Therefore, in asbestos analysis, XRD does not distinguish between fibrous and nonfibrous forms of the serpentine and amphibole minerals (Table 2-6). However, when used in conjunction with methods such as PLM or AEM, XRD techniques can provide a reliable analytical method for the identification and characterization of asbestiform minerals in bulk materials.

For **qualitative** analysis by XRD methods, samples should initially be scanned over limited diagnostic peak regions for the serpentine (~7.4 Å) and amphibole (8.2-8.5 Å) minerals (Table 2-7). Standard slow-scanning methods for bulk sample analysis may be used for materials shown by PLM to contain significant amounts of asbestos (>5 percent). Detection of minor or trace amounts of asbestos may require special sample preparation and step-scanning analysis. All samples that exhibit diffraction peaks in the diagnostic regions for asbestiform minerals should be submitted to a full (5°-60° 2θ; 1° 2θ/min) qualitative XRD scan, and their diffraction patterns should be compared with standard reference powder diffraction patterns[3] to verify initial peak assignments and to identify possible matrix interferences when subsequent quantitative analysis will be performed.

Accurate **quantitative** analysis of asbestos in bulk samples by XRD is critically dependent on particle size distribution, crystallite size, preferred orientation and matrix absorption effects, and comparability of standard reference and sample materials. The most intense diffraction peak that has been shown to be free from interference by prior qualitative XRD analysis should be selected for quantitation of each asbestiform mineral. A "thin-layer" method of analysis[5,6] can be used in which, subsequent to comminution of the bulk material to $\sim 10\ \mu m$ by suitable cryogenic milling techniques, an accurately known amount of the sample is deposited on a silver membrane filter. The mass of asbestiform material is determined by measuring the integrated area of the selected diffraction peak using a step-scanning mode, correcting for matrix absorption effects, and comparing with suitable calibration standards. Alternative "thick-layer" or bulk methods[7,8] are commonly used for **semi-quantitative analysis.**

TABLE 2-6. THE ASBESTOS MINERALS AND THEIR NONASBESTIFORM ANALOGS

Asbestiform	Nonasbestiform	Chemical Abstract Service No.
Serpentine		
Chrysotile	Antigorite, lizardite	12001-29-5
Amphibole		
Anthophyllite asbestos	Anthophyllite	77536-67-5
Cummingtonite-grunerite asbestos (Amosite)	Cummingtonite-grunerite	12172-73-5
Crocidolite	Riebeckite	12001-28-4
Tremolite asbestos	Tremolite	77536-68-6
Actinolite asbestos	Actinolite	77536-66-4

TABLE 2-7. PRINCIPAL LATTICE SPACINGS OF ASBESTIFORM MINERALS[1]

Minerals	Principal d-spacings (Å) and relative intensities			JCPDS Powder diffraction file[2] number
Chrysotile (Serpentine)	7.31_{100} 7.36_{100} 7.10_{100}	3.65_{70} 3.66_{80} 2.33_{80}	4.57_{50} 2.45_{65} 3.55_{70}	21-543[3] 25-645 22-1162 (theoretical)
Amosite (Grunerite)	8.33_{100} 8.22_{100}	3.06_{70} 3.060_{85}	2.756_{70} 3.25_{70}	17-745 (nonfibrous) 27-1170 (UICC)
Anthophyllite	3.05_{100} 3.06_{100}	3.24_{60} 8.33_{70}	8.26_{55} 3.23_{50}	9-455 16-401 (synthetic)
Crocidolite (Riebeckite)	8.35_{100} 8.40_{100}	3.10_{55} 3.12_{55}	2.720_{35} 2.726_{40}	27-1415 (UICC) 19-1061
Actinolite	2.72_{100}	2.54_{100}	3.40_{80}	25-157
Tremolite	8.38_{100} 2.706_{100} 3.13_{100}	3.12_{100} 3.14_{95} 2.706_{60}	2.705_{90} 8.43_{40} 8.44_{40}	13-437[3] 20-1310[3] (synthetic) 23-666 (synthetic mixture w/richterite)

1. This information is intended as a guide only. Complete powder diffraction data, including mineral type and source, should be referred to ensure comparability of sample and reference materials where possible. Additional precision XRD data on amosite, crocidolite, tremolite and chrysotile are available from the U.S. Bureau of Mines, Reference 4.

2. From Reference 3

3. Fibrosity questionable

This XRD method is applicable as a confirmatory method for identification and quantitation of asbestos in bulk material samples that have undergone prior analysis by PLM or other optical methods.

2.4.2 Range and Sensitivity

The range and sensitivity of the method have not been determined. They will be variable and dependent upon many factors, including matrix effects (absorption and interferences), diagnostic reflections selected and their relative intensities, preferred orientation, and instrumental limitations. A detection limit of one percent is feasible given certain sample characteristics.

2.4.3 Limitations

2.4.3.1 Interferences

Since the asbestiform and nonasbestiform analogs of the serpentine and amphibole minerals (Table 2-7) are indistinguishable by XRD techniques unless special sample preparation techniques and instrumentation are used,[9] the presence of nonasbestiform serpentines and amphiboles in a sample will pose severe interference problems in the identification and quantitative analysis of their asbestiform analogs.

The use of XRD for identification and quantitation of asbestiform minerals in bulk samples may also be limited by the presence of other interfering materials in the sample. For naturally-occurring materials, the commonly associated asbestos-related mineral interferences can usually be anticipated. However, for fabricated materials, the nature of the interferences may vary greatly (Table 2-8) and present more serious problems in identification and quantitation.[10] Potential interferences are summarized in Table 2-9 and include the following:

- **Chlorite** has major peaks at 7.19 Å and 3.58 Å that interfere with both the primary (7.31 Å) and secondary (3.65 Å) peaks for serpentine (chrysotile). Resolution of the primary peak to give good quantitative results may be possible when a step-scanning mode of operation is employed.

- **Vermiculite** has secondary peaks at 7.14 Å and 3.56 Å that could interfere with the primary peak (7.31 Å) and a secondary peak (3.65 Å) of serpentine (chrysotile).

TABLE 2-8. COMMON CONSTITUENTS IN BUILDING MATERIAL
(From Ref. 10)

A. Insulation Materials

Chrysotile
Amosite
Crocidolite
*Rock wool
*Slag wool
*Fiber glass
Gypsum ($CaSO_4 \cdot 2H_2O$)
Vermiculite (micas)
*Perlite
Clays (kaolin)
*Wood pulp
*Paper fibers (talc, clay carbonate filters)
Calcium silicates (synthetic)
Opaques (chromite, magnetite inclusions in serpentine)
Hematite (inclusions in "amosite")
Magnesite
*Diatomaceous earth

B. Flooring Materials

Calcite
Dolomite
Titanium Oxide
Quartz
Antigorite
Chrysotile
Anthophyllite
Tremolite
*Organic binders
Talc
Wollastonite

C. Spray Finishes or Paints

Bassanite
Carbonate minerals (calcite, dolomite, vaterite)
Talc
Tremolite
Anthophyllite
Serpentine (including chrysotile)
Amosite
Crocidolite
*Mineral wool
*Rock wool
*Slag wool
*Fiber glass
Clays (kaolin)
Micas
Chlorite
Gypsum
Quartz
*Organic binders and thickeners
Hydromagnesite
Wollastonite
Opaques (chromite, magnetite inclusion in serpentine)
Hematite (inclusions in "amosite")

D. Cementitious Materials

Chrysotile
Amosite
Crocidolite
Micas
Fiber glass
Cellulose
Animal hair
Quartz
Gypsum
Calcite
Dolomite
Calcium silicates

E. Roofing Materials

Chrysotile
Cellulose
Fiber glass
Mineral Wool
Asphalt
Quartz
Talc
Micas

* Amorphous materials--contribute only to overall scattered radiation and increased background radiation.

TABLE 2-9 INTERFERENCES IN XRD ANALYSIS OF ASBESTIFORM MINERALS

Asbestiform Mineral	Primary diagnostic peaks (approximate d spacings in Å)	Interference
Serpentine Chrysotile	7.3	Nonasbestiform serpentines, (antigorite, lizardite), chlorite, vermiculite, sepiolite, kaolinite, gypsum
	3.7	Nonasbestiform serpentines (antigorite, lizardite), chlorite, vermiculite, halloysite, cellulose
Amphibole Amosite (Grunerite) Anthophyllite Crocidolite (Riebeckite) Tremolite Actinolite	3.1	Nonasbestiform amphiboles (grunerite-cummingtonite, anthophyllite, riebeckite, tremolite), mutual interferences, talc, carbonates
	8.3	Nonasbestiform amphiboles (grunerite-cummingtonite, anthophyllite, riebeckite, tremolite), mutual interferences

- **Sepiolite** produces a peak at 7.47 Å which could interfere with the primary peak (7.31 Å) of serpentine (chrysotile).

- **Halloysite** has a peak at 3.63 Å that interferes with the secondary (3.65 Å) peak for serpentine (chrysotile).

- **Kaolinite** has a major peak at 7.15 Å that may interfere with the primary peak of serpentine (chrysotile) at 7.31 Å when present at concentrations of >10 percent. However, the secondary serpentine (chrysotile) peak at 3.65 Å may be used for quantitation.

- **Gypsum** has a major peak at 7.5 Å that overlaps the 7.31 Å peak of serpentine (chrysotile) when present as a major sample constituent. This may be removed by careful washing with distilled water, or by heating to 300°C to convert gypsum to plaster of paris (bassanite).

- **Cellulose** has a broad peak that partially overlaps the secondary (3.65 Å) serpentine (chrysotile) peak.[8]

- Overlap of major diagnostic peaks of the amphibole minerals, grunerite (amosite), anthophyllite, riebeckite (crocidolite), and tremolite, at approximately 8.3 Å and 3.1 Å causes mutual interference when these minerals occur in the presence of one another. In some instances adequate resolution may be attained by using step-scanning methods and/or by decreasing the collimator slit width at the x-ray port.

- **Carbonates** may also interfere with quantitative analysis of the amphibole minerals grunerite (amosite), anthophyllite, riebeckite (crocidolite), and tremolite-actinolite. Calcium carbonate ($CaCO_3$) has a peak at 3.035 Å that overlaps major amphibole peaks at approximately 3.1 Å when present in concentrations of >5 percent. Removal of carbonates with a dilute acid wash is possible; however, the time in acid should be no more than 20 minutes to preclude any loss of chrysotile.[11]

- A major **talc** peak at 3.12 Å interferes with the primary tremolite peak at this same position and with secondary peaks of actinolite (3.14 Å), riebeckite (crocidolite) (3.10 Å), grunerite (amosite) (3.06 Å), and anthophyllite (3.05 Å). In the presence of talc, the major diagnostic peak at approximately 8.3 Å should be used for quantitation of these asbestiform minerals.

The problem of intraspecies and matrix interference is further aggravated by the variability of the silicate mineral powder diffraction patterns themselves, which often makes definitive identification of the asbestos minerals by comparison with standard reference diffraction patterns difficult. This variability results from alterations in the crystal lattice associated with differences in isomorphous substitution and degree of crystallinity. This is especially true for the amphiboles. These minerals exhibit a wide variety of very similar chemical compositions, resulting in diffraction patterns characterized by having major (110) reflections of the monoclinic amphiboles and (210) reflections of orthorhombic anthophyllite separated by less than 0.2 Å.[12]

2.4.3.2 Matrix Effects

If a copper x-ray source is used, the presence of iron at high concentrations in a sample will result in significant x-ray fluorescence, leading to loss of peak intensity, increased background intensity, and an overall decrease in sensitivity. This situation may be corrected by use of an x-ray source other than copper; however, this is often accompanied both by loss of intensity and by decreased resolution of closely spaced reflections. Alternatively, use of a diffracted beam monochromator will reduce background fluorescent radiation, enabling weaker diffraction peaks to be detected.

X-ray absorption by the sample matrix will result in overall attenuation of the diffracted beam and may seriously interfere with quantitative analysis. Absorption effects may be minimized by using sufficiently "thin" samples for analysis.[5,13,14] However, unless absorption effects are known to be the same for both samples and standards, appropriate corrections should be made by referencing diagnostic peak areas to an internal standard[7,8] or filter substrate (Ag) peak.[5,6]

2.4.3.3 Particle Size Dependence

Because the intensity of diffracted x-radiation is particle-size dependent, it is essential for accurate quantitative analysis that both sample and standard reference materials have similar particle size distributions. The optimum particle size (i.e., fiber length) range for quantitative analysis of asbestos by XRD has been reported to be 1 to 10 μm.[15] Comparability of sample and standard reference material particle size distributions should be verified by optical microscopy (or another suitable method) prior to analysis.

2.4.3.4 Preferred Orientation Effects

Preferred orientation of asbestiform minerals during sample preparation often poses a serious problem in quantitative analysis by XRD. A number of techniques have been developed for reducing preferred orientation effects in "thick layer" samples.[7,8,15] For "thin" samples on membrane filters, the preferred orientation effects seem to be both reproducible and favorable to enhancement of the principal diagnostic reflections of asbestos minerals, actually increasing the overall sensitivity of the method.[12,14] However, further investigation into preferred orientation effects in both thin layer and bulk samples is required.

2.4.3.5 Lack of Suitably Characterized Standard Materials

The problem of obtaining and characterizing suitable reference materials for asbestos analysis is clearly recognized. The National Institute of Standards and Technology can provide standard reference materials for chrysotile, amosite and crocidolite (SRM 1866) and anthophyllite, tremolite and actinolite (SRM 1867).

In addition, the problem of ensuring the comparability of standard reference and sample materials, particularly regarding crystallite size, particle size distribution, and degree of crystallinity, has yet to be adequately addressed. For example, Langer et al.[18] have observed that in insulating matrices, chrysotile tends to break open into bundles more frequently than amphiboles. This results in a line-broadening effect with a resultant decrease in sensitivity. Unless this effect is the same for both standard and sample materials, the amount of chrysotile in the sample will be under-estimated by XRD analysis. To minimize this problem, it is recommended that standardized matrix reduction procedures be used for both sample and standard materials.

2.4.4 Precision and Accuracy

Neither the precision nor accuracy of this method has been determined. The individual laboratory should obtain or prepare a set of calibration materials containing a range of asbestos weight percent concentrations in combination with a variety of matrix/binder materials. Calibration curves may be constructed for use in semi-quantitative analysis of bulk materials.

2.4.5 Procedure

2.4.5.1 Sampling

Samples taken for analysis of asbestos content should be collected as specified by EPA[19]

2.4.5.2 Analysis

All samples must be analyzed initially for asbestos content by PLM. XRD may be used as an additional technique, both for identification and quantitation of sample components. **Note: Asbestos is a toxic substance. All handling of dry materials should be performed in a safety-hood.**

2.4.5.2.1 Sample Preparation

The method of sample preparation required for XRD analysis will depend on: (1) the condition of the sample received (sample size, homogeneity, particle size distribution, and overall composition as determined by PLM); and (2) the type of XRD analysis to be performed (qualitative or quantitative; thin-layer or bulk).

Bulk materials are usually received as heterogeneous mixtures of complex composition with very wide particle size distributions. Preparation of a homogeneous, representative sample from asbestos-containing materials is particularly difficult because the fibrous nature of the asbestos minerals inhibits mechanical mixing and stirring, and because milling procedures may cause adverse lattice alterations.

A discussion of specific matrix reduction procedures is given below. Complete methods of sample preparation are detailed in Sections 2.4.5.3 and 2.4.5.4. Note: **All samples should be examined microscopically before and after each matrix reduction step to monitor changes in sample particle size distribution, composition, and crystallinity, and to ensure sample representativeness and homogeneity for analysis.**

2.4.5.2.2 Milling

Mechanical milling of asbestos materials has been shown to decrease fiber crystallinity, with a resultant decrease in diffraction intensity of the specimen; the degree of lattice alteration is related to the duration and type of milling process.[20-23] Therefore, all milling times should be kept to a minimum.

For **qualitative analysis**, particle size is not usually of critical importance and initial characterization of the material with a minimum of matrix reduction is often desirable to document the composition of the sample as received. Bulk samples of very large particle size (>2-3 mm) should be comminuted to ~100 μm. A mortar and pestle can sometimes be used in size reduction of soft or loosely bound materials though this may cause matting of some samples. Such samples may be reduced by cutting with a razor blade in a mortar, or by grinding in a suitable mill (e.g., a microhammer mill or equivalent). When using a mortar for grinding or cutting, the sample should be moistened with ethanol, or some other suitable wetting agent, to minimize exposure, and the procedure should be performed in a HEPA-filtered hood.

For accurate, reproducible quantitative analysis, the particle size of both sample and standard materials should be reduced to $\sim 10\ \mu m$. Dry ball milling at liquid nitrogen temperatures (e.g., Spex Freezer Mill®, or equivalent) for a maximum time of 10 minutes (some samples may require much shorter milling time) is recommended to obtain satisfactory particle size distributions while protecting the integrity of the crystal lattice.[5] Bulk samples of very large particle size may require grinding in two stages for full matrix reduction to $< 10\ \mu m$.[8,16]

Final particle size distributions should always be verified by optical microscopy or another suitable method.

2.4.5.2.3 Ashing

For materials shown by PLM to contain large amounts of cellulose or other organic materials, it may be desirable to ash prior to analysis to reduce background radiation or matrix interference. Since chrysotile undergoes dehydroxylation at temperatures between 550°C and 650°C, with subsequent transformation to forsterite,[24,25] ashing temperatures should be kept below 500°C. Use of a muffle furnace is recommended. In all cases, calibration of the furnace is essential to ensure that a maximum ashing temperature of 500°C is not exceeded (see Section 2.3).

2.4.5.2.4 Acid Washing

Because of the interference caused by gypsum and some carbonates in the detection of asbestiform minerals by XRD (see Section 2.4.3.1), it may be necessary to remove these interferences by a simple acid washing procedure prior to analysis (see Section 2.3).

2.4.5.3 Qualitative Analysis

2.4.5.3.1 Initial Screening of Bulk Material

Qualitative analysis should be performed on a representative, homogeneous portion of the sample, with a minimum of sample treatment, using the following procedure:

1. Grind and mix the sample with a mortar and pestle (or equivalent method, see Section 2.4.5.2.2) to a final particle size sufficiently small ($\sim 100\ \mu m$) to allow adequate packing into a sample holder.

2. Pack sample into a standard bulk sample holder. Care should be taken to ensure that a representative portion of the milled sample is selected for analysis. Particular care should be taken to avoid possible size segregation of the sample. (Note: Use of **back-packing method[26] for bulk sample preparation may reduce preferred orientation effects.)**

3. Mount the sample on the diffractometer and scan over the diagnostic peak regions for the serpentine (~ 7.4 Å) and amphibole (8.2-8.5 Å) minerals (see Table 2-7). The x-ray diffraction equipment should be optimized for intensity. A slow scanning speed of $1° \ 2\theta$/min is recommended for adequate resolution. Use of a sample spinner is recommended.

4. Submit all samples that exhibit diffraction peaks in the diagnostic regions for asbestiform minerals to a full qualitative XRD scan ($5°$-$60° \ 2\theta$; $1° \ 2\theta$/min) to verify initial peak assignments and to identify potential matrix interferences when subsequent quantitative analysis is to be performed.

5. Compare the sample XRD pattern with standard reference powder diffraction patterns (i.e., JCPDS powder diffraction data[3] or those of other well-characterized reference materials). Principal lattice spacings of asbestiform minerals are given in Table 2-7; common constituents of bulk insulation and wall materials are listed in Table 2-8.

2.4.5.3.2 Detection of Minor or Trace Constituents

Routine screening of bulk materials by XRD may fail to detect small concentrations ($<1\%$) of asbestos. The limits of detection will, in general, be improved if matrix absorption effects are minimized, and if the sample particle size is reduced to the optimal 1 to 10 μm range, provided that the crystal lattice is not degraded in the milling process. Therefore, in those instances when confirmation of the presence of an asbestiform mineral at very low levels is required, or where a negative result from initial screening of the bulk material by XRD (see Section 2.4.5.3.1) is in conflict with previous PLM results, it may be desirable to prepare the sample as described for quantitative analysis (see Section 2.4.5.4) and step-scan over appropriate 2θ ranges of selected diagnostic peaks (Table 2-7). Accurate transfer of the sample to the silver membrane filter is not necessary unless subsequent quantitative analysis is to be performed.

2.4.5.4 Quantitative Analysis

The proposed method for quantitation of asbestos in bulk samples is a modification of the NIOSH-recommended thin-layer method for chrysotile in air.[6] A thick-layer bulk method involving pelletizing the sample may be used for semi-quantitative analysis;[7,8] however, this method requires the addition of an internal standard, use of a specially fabricated sample press, and relatively large amounts of standard reference materials. Additional research is required to evaluate the comparability of thin- and thick-layer methods for quantitative asbestos analysis.

For quantitative analysis by thin-layer methods, the following procedure is recommended:

1. Mill and size all or a substantial representative portion of the sample as outlined in Section 2.4.5.2.2.

2. Dry at 60°C for 2 hours; cool in a desiccator.

3. Weigh accurately to the nearest 0.01 mg.

4. Samples shown by PLM to contain large amounts of cellulosic or other organic materials, gypsum, or carbonates, should be submitted to appropriate matrix reduction procedures described in Sections 2.4.5.2.3 and 2.4.5.2.4. After ashing and/or acid treatment, repeat the drying and weighing procedures described above, and determine the percent weight loss, L.

5. Quantitatively transfer an accurately weighed amount (50-100 mg) of the sample to a 1-L volumetric flask containing approximately 200 mL isopropanol to which 3 to 4 drops of surfactant have been added.

6. Ultrasonicate for 10 minutes at a power density of approximately 0.1 W/mL, to disperse the sample material.

7. Dilute to volume with isopropanol.

8. Place flask on a magnetic-stirring plate. Stir.

9. Place silver membrane filter on the filtration apparatus, apply a vacuum, and attach the reservoir. Release the vacuum and add several milliliters of isopropanol to the reservoir. Vigorously hand shake the asbestos suspension and immediately withdraw

an aliquot from the center of the suspension so that total sample weight, W_T, on the filter will be approximately 1 mg. Do not adjust the volume in the pipet by expelling part of the suspension; if more than the desired aliquot is withdrawn, discard the aliquot and repeat the procedure with a clean pipet. Transfer the aliquot to the reservoir. Filter rapidly under vacuum. Do not wash the reservoir walls. Leave the filter apparatus under vacuum until dry. Remove the reservoir, release the vacuum, and remove the filter with forceps. (Note: Water-soluble matrix interferences such as gypsum may be removed at this time by careful washing of the filtrate with distilled water. Extreme care should be taken not to disturb the sample.)

10. Attach the filter to a flat holder with a suitable adhesive and place on the diffractometer. Use of a sample spinner is recommended.

11. For each asbestos mineral to be quantitated, select a reflection (or reflections) that has (have) been shown to be free from interferences by prior PLM or qualitative XRD analysis and that can be used unambiguously as an index of the amount of material present in the sample (see Table 2-7).

12. Analyze the selected diagnostic reflection(s) by step-scanning in increments of 0.02° 2θ for an appropriate fixed time and integrating the counts. (A fixed count scan may be used alternatively; however, the method chosen should be used consistently for all samples and standards.) An appropriate scanning interval should be selected for each peak, and background corrections made. For a fixed time scan, measure the background on each side of the peak for one-half the peak-scanning time. The net intensity, I_a, is the difference between the peak integrated count and the total background count.

13. Determine the net count, I_{Ag}, of the filter 2.36 Å silver peak following the procedure in step 12. Remove the filter from the holder, reverse it, and reattach it to the holder. Determine the net count for the unattenuated silver peak, I°_{Ag} Scan times may be less for measurement of silver peaks than for sample peaks; however, they should be constant throughout the analysis.

14. Normalize all raw, net intensities (to correct for instrument instabilities) by referencing them to an external standard (e.g., the 3.34 Å peak of an α-quartz reference crystal). After each unknown is scanned, determine the net count, I°_r, of the reference specimen following the procedure in step 12. Determine the normalized intensities by dividing the peak intensities by I°_r:

$$\hat{I}_a = \frac{I_a}{I_r^\circ}, \quad \hat{I}_{Ag} = \frac{I_{Ag}}{I_r^\circ}, \text{ and } \hat{I}_{Ag}^\circ = \frac{I_{Ag}^\circ}{I_r^\circ}$$

2.4.6 Calibration

2.4.6.1 Preparation of Calibration Standards

1. Mill and size standard asbestos materials according to the procedure outlined in Section 2.4.5.2.2. **Equivalent standardized matrix reduction and sizing techniques should be used for both standard and sample materials.**

2. Dry at 100°C for 2 hours; cool in a desiccator.

3. Prepare two suspensions of each standard in isopropanol by weighing approximately 10 and 50 mg of the dry material to the nearest 0.01 mg. Transfer each to a 1-L volumetric flask containing approximately 200 mL isopropanol to which a few drops of surfactant have been added.

4. Ultrasonicate for 10 minutes at a power density of approximately 0.1 W/mL, to disperse the asbestos material.

5. Dilute to volume with isopropanol.

6. Place the flask on a magnetic stirring plate. Stir.

7. Prepare, in triplicate, a series of at least five standard filters to cover the desired analytical range, using appropriate aliquots of the 10 and 50 mg/L suspensions. For each standard, mount a silver membrane filter on the filtration apparatus. Place a few mL of isopropanol in the reservoir. Vigorously hand shake the asbestos suspension and immediately withdraw an aliquot from the center of the suspension. Do not adjust the volume in the pipet by expelling part of the suspension; if more than the desired aliquot is withdrawn, discard the aliquot and resume the procedure with a clean pipet. Transfer the aliquot to the reservoir. Keep the tip of the pipet near the surface of the isopropanol. Filter rapidly under vacuum. Do not wash the sides of the reservoir. Leave the vacuum on for a time sufficient to dry the filter. Release the vacuum and remove the filter with forceps.

2.4.6.2 Analysis of Calibration Standards

1. Mount each filter on a flat holder. Perform step scans on selected diagnostic reflections of the standards and reference specimen using the procedure outlined in Section 2.4.5.4, step 12, and the same conditions as those used for the samples.

2. Determine the normalized intensity for each peak measured, $\hat{I}^{\circ}_{std}$, as outlined in Section 2.4.5.4, step 14.

2.4.7 Calculations

For each asbestos reference material, calculate the exact weight deposited on each standard filter from the concentrations of the standard suspensions and aliquot volumes. Record the weight, w, of each standard. Prepare a calibration curve by regressing $\hat{I}^{\circ}_{std}$, on w. Poor reproducibility ($\pm$15 percent RSD) at any given level indicates problems in the sample preparation technique, and a need for new standards. The data should fit a straight-line equation.

Determine the slope, m, of the calibration curve in counts/microgram. The intercept, b, of the line with the $\hat{I}^{\circ}_{std}$ axis should be approximately zero. A large negative intercept indicates an error in determining the background. This may arise from incorrectly measuring the baseline or from interference by another phase at the angle of background measurement. A large positive intercept indicates an error in determining the baseline or that an impurity is included in the measured peak.

Using the normalized intensity, $\hat{I}_{Ag}$ for the attenuated silver peak of a sample, and the corresponding normalized intensity from the unattenuated silver peak $\hat{I}^{\circ}_{Ag}$, of the sample filter, calculate the transmittance, T, for each sample as follows:[27,28]

$$T = \frac{\hat{I}_{Ag}}{\hat{I}^{\circ}_{Ag}}$$

Determine the correction factor, f(T), for each sample according to the formula:

$$f(T) = \frac{-R(\ln T)}{1 - T^R}$$

where

$$R = \frac{\sin \theta_{Ag}}{\sin \theta_a}$$

θ_{Ag} = angular position of the measured silver peak (from Bragg's Law), and

θ_a = angular position of the diagnostic asbestos peak.

Calculate the weight, W_a, in micrograms, of the asbestos material analyzed for in each sample, using the absorption corrections:

$$W_a = \frac{\hat{I}_a f(t) - b}{m}$$

Calculate the percent composition, P_a, of each asbestos mineral analyzed for in the parent material, from the total sample weight, W_T, on the filter:

$$P_a = \frac{W_a(1 - .01L)}{W_T} \times 100$$

where

P_a = percent asbestos mineral in parent material;

W_a = mass of asbestos mineral on filter, in μg;

W_T = total sample weight on filter, in μg;

L = percent weight loss of parent material on ashing and/or acid treatment (see Section 2.4.5.4).

2.4.8 References

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2.5 Analytical Electron Microscopy

2.5.1 Applicability

Analytical electron microscopy (AEM) can often be a reliable method for the detection and positive identification of asbestos in some bulk building materials, both friable and nonfriable. The method is particularly applicable to bulk materials that contain a large amount of interfering materials that can be removed by ashing and/or dissolution and contain asbestos fibers that are not resolved by PLM techniques. Many floor tiles and plasters would be included in this type of sample. In combination with suitable specimen preparation techniques, the AEM method can also be used to quantify asbestos concentrations.

2.5.2 Range

The range is dependent on the type of bulk material being analyzed. The upper detection limit is 100%, and the lower detection limit can be as low as 0.0001% depending on the extent to which interfering materials can be separated during the preparation of AEM

specimens, the sophistication of the AEM preparation, and the amount of labor expended on AEM examination.

2.5.3 Interferences

The presence of a large amount of binder/matrix materials associated with fibers can make it difficult to positively identify fibers as asbestos. The portion of the fiber examined by either electron diffraction or energy dispersive x-ray analysis (EDXA) must be free of binder/matrix materials.

2.5.4 Precision and Accuracy

The precision and accuracy of the method have not been determined.

2.5.5 Procedures

The procedures for AEM specimen preparation depend on the data required. In analysis of floor tiles, the weighed residue after removal of the matrix components (see Section 2.3, Gravimetry) is often mostly asbestos, and the task is primarily to identify the fibers. In this situation the proportion of asbestos in the residue can be estimated by AEM and this estimate can be used to refine the gravimetric result. For many floor tiles, the final result is not very sensitive to errors in this estimation because the proportion of asbestos in the residue is very high. For samples in which this is not the case, precise measurements can be made using a quantitative AEM preparation, in which each grid opening of the specimen grid corresponds to a known weight of the original sample or of a concentrate derived from the original sample. Asbestos fibers on these grids are then identified and measured, using a fiber counting protocol which is directed towards a precise determination of mass concentration. This latter procedure is suitable for samples of low asbestos concentration, or for those in which it is not possible to remove a large proportion of the matrix material.

2.5.5.1 AEM Specimen Preparation for Semi-Quantitative Evaluation

The residual material from any ashing or dissolution procedures (see Section 2.3) used (usually trapped on a membrane filter) should be placed in a small volume of ethanol or another solvent such as acetone or isopropyl alcohol, in a disposable beaker, and dispersed by treatment in an ultrasonic bath. A small volume of this suspension (approximately 3μl) should be pipetted onto the top of a carbon-coated TEM grid. The suspension should be allowed to dry under a heat lamp. The grid is then ready for examination.

Samples that are not conducive to ashing or dissolution may also be prepared in this way for AEM analysis. A few milligrams of the sample may be ground in a mortar and pestle or milled, dispersed in ethanol or another solvent using an ultrasonic bath, and pipetted onto a grid as described previously.

2.5.5.2 AEM Specimen Preparation for Quantitative Evaluation

The objective of this preparation is to obtain a TEM grid on which a known weight of the bulk sample is represented by a known area of the TEM grid. A known weight of the bulk sample, or of the residue after extraction, should be dispersed in a known volume of distilled water. Aliquots of this dispersion should then be filtered through 0.22 μm pore-size MCE or 0.2 μm pore-size PC filters, using filtration techniques as described for analysis of water samples.[1] In order to obtain filters of appropriate particulate loading for AEM analysis, it may be necessary to perform serial dilutions of the initial dispersion. TEM grids should then be prepared from appropriately-loaded filters, using the standard methods.[2]

Determination of the mass concentration of asbestos on the TEM grids requires a different fiber counting protocol than that usually used for determination of numerical fiber concentrations. Initially, the grids should be scanned to determine the dimensions of the largest asbestos fiber or fiber bundle on the specimens. The volume of this fiber or bundle should be calculated. The magnification of the AEM should be set at a value for which the length of this fiber or bundle just fills the fluorescent screen. Asbestos fiber counting should then be continued at this magnification. The count should be terminated when the volume of the initial large fiber or bundle represents less than about 5% of the integrated volume of all asbestos fibers detected. This counting strategy ensures that the fiber counting effort is directed toward those fibers which contribute most to the mass, and permits a precise mass concentration value to be obtained.

2.5.5.2.1 Identification

To document the positive identification of asbestos in a sample, the analyst should record the following physical properties: morphology data, electron diffraction data, EDXA data, and any other distinguishing characteristics observed. For fibrous structures identified as nonasbestos, the unique physical property or properties that differentiate the material from asbestos should be recorded.

The purpose of the identification data collected is to prevent or limit false negatives and false positives. This can be accomplished by having a system for measuring and recording the d-spacings and symmetry of the diffraction patterns, determining the relative abundance of the elements detected by EDXA, and comparing these results to reference data. The laboratory should have a set of reference asbestos materials from which a set of reference diffraction patterns and x-ray spectra have been developed. Also, the laboratory should have available reference data on the crystallography and chemical composition of minerals that might analytically interfere with asbestos.

2.5.6 References

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2. Environmental Protection Agency's **Interim Transmission Electron Microscopy Analytical Methods--Mandatory and Nonmandatory--and Mandatory Section to Determine Completion of Response Actions**, Appendix A to subpart E, 40 CFR part 763.

2.6 Other Methodologies

Additional analytical methods (e.g. Scanning Electron Microscopy) may be applicable for some bulk materials. However, the analyst should take care to recognize the limitations of any analytical method chosen. Conventional SEM, for example, cannot detect small diameter fibers ($\sim < 0.2\mu m$), and cannot determine crystal structure. It is, however, very useful for observing surface features in complex particle matrices, and for determining elemental compositions.

3.0 QUALITY CONTROL/QUALITY ASSURANCE OPERATIONS- PLM

A program to routinely assess the quality of the results produced by the PLM laboratory must be developed and implemented. **Quality Control** (QC) is a system of activities whose purpose is to control the quality of the product or service so that it meets the need of the users. This also includes **Quality Assessment**, whose purpose is to provide assurance that the overall quality control is being done effectively. While the essential elements of a quality control system are described in detail elsewhere,[1,2,3,4,5,6] only several of the elements will be discussed here. **Quality Assurance (QA)** is comprised of Quality Control and Quality Assessment and is a system of activities designed to provide assurance that a product or service meets defined standards of quality.

The purpose of the Quality Assurance program is to minimize failures in the analysis of materials prior to submitting the results to the client. Failures in the analysis of asbestos materials include false positives, false negatives, and misidentification of asbestos types. False positives result from identification or quantitation errors. False negatives result from identification, detection, or quantitation errors.

For the stereomicroscopic and PLM techniques, the quality control procedures should characterize the accuracy and precision of both individual analysts and the techniques. Analysts should demonstrate their abilities on calibration materials, and also be checked routinely on the analysis of unknowns by comparison with results of a second analyst. The limitations of the stereomicroscopic and PLM techniques can be determined by using a second analytical technique, such as gravimetry, XRD, or AEM. For example, stereomicroscopic and PLM techniques can fail in the analysis of floor tiles because the asbestos fibers in the sample may be too small to be resolved by light microscopy. An XRD or AEM analysis is not subject to the same limitations, and may indicate the presence of asbestos in the sample.

The accuracy, precision, and detection limits of all analytical techniques described in this method are dependent on the type of sample (matrix components, texture, etc.), on the preparation of the sample (homogeneity, grain size, etc.), and the specifics of the method (number of point counts for PLM, mass of sample for gravimetry, counting time for XRD, etc.). These should be kept in mind when designing quality control procedures and characterizing performance, and are variables that must be tracked in the quality assurance system.

3.1 General Considerations

3.1.1 Training

Of paramount importance in the successful use of this or any other analytical method is the well-trained analyst. It is highly recommended that the analyst have completed course work in optical mineralogy on the collegiate level. That is not to say that others cannot successfully use this method, but the classification error rate[7] may, in some cases, be directly attributable to level of training. In addition to completed course work in optical mineralogy, specialized course work in PLM and asbestos identification by PLM is desirable. Experience is as important as education. A good laboratory training program can be used in place of course work. Analysts that are in training and not yet fully qualified should have all analyses checked by a qualified analyst before results are released. A QC Plan for asbestos identification would be considered incomplete without a detailed description of the analyst training program, together with detailed records of training for each analyst.

3.1.2 Instrument Calibration and Maintenance

Microscope alignment checks (alignment of the polarizer at 90° with respect to the analyzer, and coincident with the cross-lines, proper orientation of the slow vibration direction of the Red I compensator plate, image of the field diaphragm focussed in the plane of the specimen, centering of the central dispersion staining stop, etc.) should be performed with sufficient frequency to ensure proper operations. Liquids used for refractive index determination and those optionally used for dispersion staining should have periodic refractive index checks using a refractometer or known refractive index solids. These calibrations must be documented.

Microscopes and ancillary equipment should be maintained daily. It is recommended that at least once per year each microscope be thoroughly cleaned and re-aligned by a professional microscope service technician. Adequate inventories of replaceable parts (illumination lamps, etc.) should be established and maintained. All maintenance must be documented.

3.2 Quality Control of Asbestos Analysis

3.2.1 Qualitative Analysis

All analysts must be able to correctly identify the six regulated asbestos types (chrysotile, amosite, crocidolite, anthophyllite, actinolite, and tremolite) using combined stereomicroscopic and PLM techniques. Standards for the six asbestos types listed are available from NIST, and should be used to train analysts in the measurement of optical properties and identification of asbestos. These materials can also be used as identification standards for XRD and AEM.

Identification errors between asbestos types (e.g. reporting amosite when tremolite is present) implies that the analyst cannot properly determine optical properties and is relying on morphology as the identification criteria. This is not acceptable. Each analyst in the lab should prove his or her proficiency in identifying the asbestos types; this can be checked through use of calibration materials (NVLAP proficiency testing materials, materials characterized by an independent technique, and synthesized materials) and by comparing results with another analyst. The identification of all parameters (e.g. refractive indices, birefringence, sign of elongation, etc.) leading to the identification should fall within control limits determined by the laboratory. In addition, a subset of materials should be analyzed using another technique to confirm the analysis.

As discussed earlier, the qualitative analysis is dependent upon matrix and asbestos type and texture. Therefore, the quality assurance system should monitor for samples that are difficult to analyze and develop additional or special steps to ensure accurate characterization of these materials. When an analyst is found to be out of the control limits defined by the laboratory, he or she should undergo additional training and have confirmatory analyses performed on all samples until the problem has been corrected.

3.2.2 Quantitative Analysis

The determination of the amount of asbestos in a sample can be accomplished using the various techniques outlined in this method. The mandatory stereomicroscopic and PLM examinations provide concentrations in terms of volume, area, or weight, depending upon the calibration procedure. Gravimetric and quantitative XRD techniques result in concentrations in units of weight percent. Specific guidelines for determining accuracy and precision using these techniques are provided in the appropriate sections of this method. In general, however, the accuracy of any technique is determined through analysis of calibration materials which are characterized by multiple independent techniques in order to provide an unbiased value for the analyte (asbestos) in question. The precision of any technique is determined by multiple analyses of the sample. The analyst is the detector for stereomicroscopic and PLM techniques, as opposed to gravimetric and XRD techniques, and therefore must be calibrated as an integral part of the procedure.

As in the qualitative analysis, the laboratory should determine its accuracy and precision for quantitative asbestos analysis according to the type of material analyzed and the technique used for analysis. For example, the laboratory may determine that its analysts have a problem with calibrated area estimates of samples containing cellulose and chrysotile and therefore needs to make or find special calibration materials for this class of sample.

Calibration materials for quantitative analysis of asbestos are available through the Bulk Asbestos NVLAP as proficiency testing materials for those laboratories enrolled in NVLAP. In a report provided following a test round, the concentration of asbestos in each sample is given in weight percent with 95%/95% tolerance limits, along with a description of the major matrix components. Materials from other round robin and quality assurance programs for asbestos analysis may not have been analyzed by independent techniques; the concentrations may represent consensus PLM results that could be significantly biased. Therefore, values from these programs should <u>not</u> be used as calibration materials for quantitative analysis.

Calibration materials for quantitative analysis can also be synthesized by mixing asbestos and appropriate matrix materials, as described in Appendix C of this method. These materials are usually simplifications of "real world" samples; therefore the accuracy and precision determined from analysis of these materials are probably ideal.

Limits on permissible analytical variability must be established by the laboratory prior to QC implementation. It is recommended that a laboratory initially be at 100% quality control (all samples reanalyzed.) The proportion of quality control samples can later be lowered gradually, as control indicates, to a minimum of 10%. Quantitative results for standards including the mean and error estimate (typically 95% confidence or tolerance intervals) should be recorded. Over time these data can be used to help determine control limits for quality control charts.

The establishment and use of control charts is extensively discussed elsewhere in the literature.[1,2,3,4,5] Several cautions are in order:

- Control charts are based on the assumption that the data are distributed normally. Using rational subgrouping, the means of the subgroups are approximately normally distributed, irrespective of the distribution of the individual values in the subgroups. Control charts for asbestos analysis are probably going to be based on individual measurements, not rational subgroups. Check the data for normality before proceeding with the use of control charts. Ryan[8] suggests a minimum of 50 analyses before an attempt is made to establish control limits. However, for this analysis, consider setting "temporary" limits after accumulating 20-30 analyses of the sample.

- Include <u>both</u> prepared slides as well as bulk samples in your reference inventory.

- Make certain that sample quantities are sufficient to last, and that the act of sampling will not alter the composition of the reference sample.

Data on analytical variability can be obtained by having analysts repeat their analyses of samples and also by having different analysts analyze the same samples.

3.3 Interlaboratory Quality Control

The establishment and maintenance of an interlaboratory QC program is fundamental to continued assurance that the data produced within the laboratory are of consistent high quality. Intralaboratory programs may not be as sensitive to accuracy and precision error, especially if the control charts (see Section 3.2.2) for all analysts in the laboratory indicate small percent differences. A routine interlaboratory testing program will assist in the detection of internal bias and analyses may be performed more frequently than proficiency testing. Arrangements should be made with at least two (preferably more) other laboratories that conduct asbestos identification by PLM. Samples (the number of which is left to the participating laboratories, but at least 4-10) representing the types of samples and matrices routinely submitted to the lab for analysis should be exchanged with sufficient frequency to determine intralaboratory bias. Both reference slides and bulk samples should be used. Results of the interlaboratory testing program should be evaluated by each of the participating laboratories and corrective actions, if needed, identified and implemented. Since quantitation problems are more pronounced at low concentrations ($\leq$ 5%), it would be prudent to include approximately 30-50% from this concentration range in the sample selection process.

3.4 Performance Audits

Performance audits are independent quantitative assessments of laboratory performance. These audits are similar to the interlaboratory QC programs established between several laboratories, but with a much larger cohort (the EPA Asbestos Bulk Sample Analysis Quality Assurance Program had as many as 1100 participating laboratories). Participation in this type of program permitted assessment of performance through the use of "consensus" test materials, and served to assist in assessing the bias relative to individual interlaboratory, as well as intralaboratory programs. Caution should be exercised in the use of "consensus" quantitation results, as they are likely to be significantly responsible for the propagation of high bias in visual estimates. The current NIST/NVLAP[9] for bulk asbestos laboratories (PLM) does not use concensus quantitation results. Results are reported in weight percent with a 95% tolerance interval. The American Industrial Hygiene Association (AIHA)[10] also conducts a proficiency testing program for bulk asbestos laboratories. Quantitation results for this program are derived from analyses by two reference laboratories and PLM, XRD and gravimetric analysis performed by Research Triangle Institute.

3.5 Systems Audits

Where performance audits are quantitative in nature, systems audits are qualitative. Systems audits are assessments of the laboratory quality system as specified in the Laboratory

Quality Assurance Manual. Such an audit might consist of an evaluation of some facet of the QA Manual, or the audit may be larger in scope. For example, the auditor might request specific laboratory data sheets which will be evaluated against written procedures for data recording in the laboratory. Or, the auditor might request air monitoring or contamination control data to review for frequency of sampling, analysis methodology, and/or corrective actions taken when problems were discovered. The audit report should reflect the nature of the audit as well as the audit results. Any recommendations for improvement should also be reflected in such a report.

3.6 References

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3. Taylor, J.R., **Quality Control Systems,** McGraw Hill, Inc., 1989.

4. Ratliff, T.A., **The Laboratory Quality Assurance System,** Van Nostrand Reinhold, 1990.

5. Taylor, J.K., **Quality Assurance of Chemical Measurements,** Lewis Publishers, 1987.

6. Bulk Asbestos Handbook, National Institute of Standards and Technology, National Voluntary Laboratory Accreditation Program, NISTIR 88-3879, October 1988.

7. Harvey, B.W., "Classification and Identification Error Tendencies in Bulk Insulation Proficiency Testing Materials," **American Environmental Laboratory, 2**(2), 4/90, pp. 8-14.

8. Ryan, T.P., **Statistical Techniques for Quality Improvement,** John Wiley & Sons, Inc., New York, 1989.

9. National Institute of Standards & Technology (NIST) National Voluntary Laboratory Accreditation Program (NVLAP), Building 411, Room A124, Gaithersburg, MD 20899, telephone (301) 975-4016.

10. American Industrial Hygiene Association (AIHA), 2700 Prosperity Avenue, Suite 250, Fairfax, VA 22031, (703) 849-8888.

APPENDIX A

Glossary Of Terms

APPENDIX A. GLOSSARY OF TERMS

Accuracy - The degree of agreement of a measured value with the true or expected value.

Anisotropic - Refers to substances that have more than one refractive index (e.g. are birefringent), such as nonisometric crystals, oriented polymers, or strained isotropic substances.

Asbestiform (morphology) - Said of a mineral that is like asbestos, i.e., crystallized with the habit of asbestos. Some asbestiform minerals may lack the properties which make asbestos commercially valuable, such as long fiber length and high tensile strength. With the light microscope, the asbestiform habit is generally recognized by the following characteristics:

- Mean aspect ratios ranging from 20:1 to 100:1 or higher for fibers longer than 5μm. Aspect ratios should be determined for <u>fibers, not bundles</u>.
- Very thin fibrils, usually less than 0.5 micrometers in width, and
- Two or more of the following:
  - Parallel fibers occurring in bundles,
  - Fiber bundles displaying splayed ends,
  - Matted masses of individual fibers, and/or
  - Fibers showing curvature

These characteristics refer to the <u>population of fibers</u> as observed in a bulk sample. It is not unusual to observe occasional particles having aspect ratios of 10:1 or less, but it is unlikely that the asbestos component(s) would be dominated by particles (individual fibers) having aspect ratios of <20:1 for fibers longer than 5μm. If a sample contains a fibrous component of which most of the fibers have aspect ratios of <20:1 and that do not display the additional asbestiform characteristics, by definition the component should not be considered asbestos.

Asbestos - A commercial term applied to the asbestiform varieties of six different minerals. The asbestos types are chrysotile (asbestiform serpentine), amosite (asbestiform grunerite), crocidolite (asbestiform riebeckite), and asbestiform anthophyllite, asbestiform tremolite, and asbestiform actinolite. The properties of asbestos that caused it to be widely used commercially are: 1) its ability to be separated into long, thin, flexible fibers; 2) high tensile strength; 3) low thermal and electrical conductivity; 4) high mechanical and chemical durability, and 5) high heat resistance.

Becke Line - A band of light seen at the periphery of a specimen when the refractive indices of the specimen and the mounting medium are different; it is used to determine refractive index.

Bias - A systematic error characterized by a consistent (non-random) measurement error.

Binder - With reference to a bulk sample, a component added for cohesiveness (e.g. plaster, cement, glue, etc.).

Birefringence - The numerical difference between the maximum and minimum refractive indices of an anisotropic substance. Birefringence may be estimated, using a Michel-Levy chart, from the interference colors observed under crossed polarizers. Interference colors are also dependent on the orientation and thickness of the grain, and therefore are used qualitatively to determine placement in one of the four categories listed below.

Qualitative	Quantitative(N-n)
none	0.00 or isotropic
low	≤0.010
moderate	0.011-0.050
high	>0.050

Bulk Sample - A sample of building material taken for identification and quantitation of asbestos. Bulk building materials may include a wide variety of friable and nonfriable materials.

Bundle - Asbestos structure consisting of several fibers having a common axis of elongation.

Calibration Materials - Materials, such as known weight % standards, that assist in the calibration of microscopists in terms of ability to quantitate the asbestos content of bulk materials.

Color - The color of a particle or fiber when observed in plane polarized light.

Compensator - A device with known, fixed or variable retardation and vibration direction used for determining the degree of retardation (hence the thickness or value of birefringence) in an anisotropic specimen. It is also used to determine the sign of elongation of elongated materials. The most common compensator is the first-order red plate (530-550nm retardation).

Control Chart - A graphical plot of test results with respect to time or sequence of measurement, together with limits within which they are expected to lie when the system is in a state of statistical control.

Detection Limit - The smallest concentration/amount of some component of interest that can be measured by a single measurement with a stated level of confidence.

Dispersion Staining (focal masking) - An optical means of imparting apparent or virtual color to transparent substances by the use of stops in the objective back focal plane; ir it is used to determine refractive indices.

Error - Difference between the true or expected value and the measured value of a quantity or parameter.

Extinction - The condition in which an anisotropic substance appears dark when observed between crossed polars. This occurs when the vibration directions in the specimen are parallel to the vibration directions in the polarizer and analyzer. Extinction may be complete or incomplete; common types include parallel, oblique, symmetrical and undulose.

Extinction Angle - For fibers, the angle between the extinction position and the position at which the fiber is parallel to the polarizer or analyzer privileged directions.

Fiber - With reference to asbestiform morphology, a structure consisting of one or more fibrils.

Fibril - The individual unit structure of fibers.

Friable - Refers to the cohesiveness of a bulk material, indicating that it may be crumbled or disaggregated by hand pressure.

Gravimetry - Any technique in which the concentration of a component is determined by weighing. As used in this document, it refers to measurement of asbestos-containing residues after sample treatment by ashing, dissolution, etc.

Homogeneous - Uniform in composition and distribution of all components of a material, such that multiple subsamples taken for analysis will contain the same components in approximately the same relative concentrations.

Heterogeneous - Lacking uniformity in composition and/or distribution of material; components not uniform. Does not satisfy the conditions stated for homogenous; e.g., layered or in clumps, very coarse grained, etc.

Isotropic - Refers to substances that have a single refractive index such as unstrained glass, un-oriented polymers and unstrained substances in the isometric crystal system.

Lamda Zero (λ_0) - The wavelength (λ_0) of the dispersion staining color shown by a specimen in a medium; both the specimen and medium have the same refractive index at that wavelength.

Matrix - Nonasbestos, nonbinder components of a bulk material. Includes such components as cellulose, fiberglass, mineral wool, mica, etc.

Michel-Levy Scale of Retardation colors - A chart plotting the relationship between birefringence, retardation and thickness of anisotropic substances. Any one of the three variables can be determined if the other two are known.

Morphology - The structure and shape of a particle. Characterization may be descriptive (platy, rod-like, acicular, etc) or in terms of dimensions such as length and diameter (see asbestiform).

Pleochroism - The change in color or hue of colored anisotropic substance when rotated relative to the vibration direction of plane polarized light.

Point Counting - A technique used to determine the relative projected areas occupied by separate components in a microscope slide preparation of a sample. For asbestos analysis, this technique is used to determine the relative concentrations of asbestos minerals to nonasbestos sample components.

Polarization Colors - Interference colors displayed by anisotropic substances between two polarizers. Birefringence, thickness and orientation of the material affect the colors and their intensity.

Precision - The degree of mutual agreement characteristic of independent measurements as the result of repeated application of the process under specified conditions. It is concerned with the variability of results.

Reference Materials - Bulk materials, both asbestos-containing and nonasbestos-containing, for which the components are well-documented as to identification and quantitation.

Refractive Index (index of refraction) - The ratio of the velocity of light in a vacuum relative to the velocity of light in a medium. It is expressed as n and varies with wavelength and temperature.

Sign of Elongation - Referring to the location of the high and low refractive indices in an elongated anisotropic substance, a specimen is described as positive when the higher refractive index is lengthwise (length slow), and as negative when the lower refractive index is lengthwise (length fast).

Standard Reference Material (SRM) - A reference material certified and distributed by the National Institute of Standards and Technology.

Visual Estimate - An estimation of concentration of asbestos in a sample as compared to the other sample components. This may be a volume estimate made during stereomicroscopic examination and/or a projected area estimation made during microscopic (PLM) examination.

APPENDIX B

Apparatus For Sample Preparation And Analysis

B1.0 INTRODUCTION

The following lists the apparatus and materials required and suggested for the methods of sample preparation and analysis described in the test method.[1,2,3]

B2.0 STEREOMICROSCOPIC EXAMINATION

The following are suggested for routine stereomicroscopic examination.

- HEPA-filtered hood or class 1 biohazard hood, negative pressure
- Microscope: binocular microscope, preferably stereoscopic, 5-60X magnification (approximate)
- Light source: incandescent or fluorescent
- Tweezers, dissecting needles, scalpels, probes, etc. (for sample manipulation)
- Glassine paper, glass plates, weigh boats, petri dishes, watchglasses, etc. (sample containers)

The following are suggested for sample preparation.

- Mortar and pestle, silica or porcelain-glazed
- Analytical balance (readability less than or equal to one milligram) (optional)
- Mill or blender (optional)

B3.0 POLARIZED LIGHT MICROSCOPY

The laboratory should be equipped with a polarized light microscope (preferably capable of Köhler or Köhler-type illumination if possible) and accessories as described below.

- Ocular(s) binocular or monocular with cross hair reticle, or functional equivalent, and a magnification of at least 8X
- 10X, 20X, and 40X objectives, (or similar magnification)

- Light source (with optional blue "day-light" filter)

- 360-degree rotatable stage

- Substage condenser with iris diaphragm

- Polarizer and analyzer which can be placed at 90 degrees to one another, and can be calibrated relative to the cross-line reticle in the ocular.

- Accessory slot for wave plates and compensators (or demonstrated equivalent).

- Wave retardation plate (Red I compensator) with approximately 550 nanometer retardation, and with known slow and fast vibration directions.

- Dispersion staining objective or a demonstrated equivalent. (optional)

- Monochromatic filter (n_D), or functional equivalent. (optional)

In addition, the following equipment, materials and reagents are required or recommended.[1]

- NIST traceable standards for the major asbestos types (NIST SRM 1866 and 1867)

- Class I biohazard hood or better (see "Note", Section 2.2.5)

- Sampling utensils (razor knives, forceps, probe needles, etc.)

- Microscope slides and cover slips

- Mechanical Stage

- Point Counting Stage (optional)

- Refractive index liquids: 1.490-1.570, 1.590-1.720 in increments of less than or equal to 0.005; high dispersion, (HD) liquids are optional; however, if using dispersion staining, HD liquids are recommended.

- Mortar and pestle

- Distilled water

- HCl, ACS reagent grade concentrated

- Muffle furnace (optional)
- Mill or blender (optional)
- Beakers and assorted glassware (optional)
- Other reagents (tetrahydrofuran, amyl acetate, acetone, sodium hexametaphosphate, etc.) (optional)

B4.0 GRAVIMETRY

The following equipment, materials, and reagents are suggested.

- Scalpels
- Crucibles, silica or porcelain-glazed, with lids
- Muffle furnace - temperature range at least to 500°C, temperature stable to $\pm$ 10°C, temperature at sample position calibrated to $\pm$ 10°C
- Filters, 0.4 μm pore size polycarbonate
- Petri dishes
- Glass filtration assembly, including vacuum flask, water aspirator, and/or air pump
- Analytical balance, readable to 0.001 gram
- Mortar and pestle, silica or porcelain-glazed
- Heat lamp or slide warmer
- Beakers and assorted glassware
- Centrifuge, bench-top
- Class I biohazard hood or better
- Bulb pipettes
- Distilled water
- HCl, reagent-grade concentrated

- Organic solvents (tetrahydrofuran, amyl acetate,etc)
- Ultrasonic bath

B5.0 X-RAY DIFFRACTION

Sample Preparation

Sample preparation apparatus requirements will depend upon the sample type under consideration and the kind of XRD analysis to be performed.

- Mortar and pestle: agate or porcelain
- Razor blades
- Sample mill: SPEX, Inc., freezer mill or equivalent
- Bulk sample holders
- Silver membrane filters: 25-mm diameter, 0.45-μm pore size. Selas Corp. of America, Flotronics Div., 1957 Pioneer Road, Huntington Valley, PA 19006
- Microscope slides
- Vacuum filtration apparatus: Gelman No. 1107 or equivalent, the side-arm vacuum flask
- Microbalance
- Ultrasonic bath or probe: Model W140, Ultrasonics, Inc., operated at a power density of approximately 0.1 W/mL, or equivalent
- Volumetric flasks: 1-L volume
- Assorted pipets
- Pipet bulb
- Nonserrated forceps
- Polyethylene wash bottle
- Pyrex beakers: 50-mL volume

- Desiccator
- Filter storage cassettes
- Magnetic stirring plate and bars
- Porcelain crucibles
- Muffle furnace or low temperature asher
- Class 1 biohazard hood or better

Sample Analysis

Sample analysis requirements include an x-ray diffraction unit, equipped with:

- Constant potential generator; voltage and mA stabilizers
- Automated diffractometer with step-scanning mode
- Copper target x-ray tube: high intensity; fine focus, preferably
- X-ray pulse height selector
- X-ray detector (with high voltage power supply): scintillation or proportional counter
- Focusing graphite crystal monochromator; or nickel filter (if copper source is used, and iron fluorescence is not a serious problem)
- Data output accessories:
  - Strip chart recorder
  - Decade scaler/timer
  - Digital printer

  or

  - PC, appropriate software and Laser Jet Printer
- Sample spinner (optional)
- Instrument calibration reference specimen: α-quartz reference crystal (Arkansas quartz standard, #180-147-00, Philips Electronics Instruments, Inc., 85 McKee Drive, Mahwah, NJ 07430) or equivalent.

Reagents, etc.

Reference Materials - The list of reference materials below is intended to serve as a guide. Every attempt should be made to acquire pure reference materials that are comparable to sample materials being analyzed.

- Chrysotile: UICC Canadian, NIST SRM 1866 (UICC reference material available from: UICC, MRC Pneumoconiosis Unit, Llandough Hospital, Penarth, Glamorgan, CF61XW, UK); (NIST Standard Reference Materials available from the National Institute of Standards and Technology, Office of Reference Standards, Gaithersburg, MD 20899)

- Crocidolite: UICC, NIST SRM 1866.

- "Amosite": UICC, NIST SRM 1866.

- Anthophyllite-Asbestos: UICC, NIST SRM 1867

- Tremolite Asbestos: Wards Natural Science Establishment, Rochester, NY; Cyprus Research Standard, Cyprus Research, 2435 Military Ave., Los Angeles, CA 900064 (washed with dilute HCl to remove small amount of calcite impurity); Indian tremolite, Rajasthan State, India; NIST SRM 1867.

- Actinolite Asbestos: NIST SRM 1867

Adhesive - Tape, petroleum jelly, etc. (for attaching silver membrane filters to sample holders).

Surfactant - 1 Percent aerosol OT aqueous solution or equivalent.

Isopropanol - ACS Reagent Grade.

B6.0 ANALYTICAL ELECTRON MICROSCOPY

AEM equipment requirements will not be discussed in this document; it is suggested that equipment requirements stated in the AHERA regulations be followed. Additional information may be found in the NVLAP Program Handbook for Airborne Asbestos Analysis.[3]

The following additional materials and equipment are suggested:

- Analytical balance, readable to 0.001 gram
- Ultrasonic bath
- Glass filtration assembly (25mm), including vacuum flask and water aspirator
- Mixed cellulose ester (MCE) filters (0.22μm pore size) or 0.2μm pore size polycarbonate filters
- MCE backing filters (5μm pore size)
- Silica mortar and pestle
- Beakers - glass and disposable
- Pipettes, disposable, 1,5, and 10 ml

B7.0 REFERENCES

1. National Institute of Standards and Technology (NIST) National Voluntary Laboratory Accreditation Program (NVLAP) Bulk Asbestos Handbook, NISTIR 88-3879, 1988.

2. **Interim Method for the Determination of Asbestos in Bulk Insulation Samples**, U.S. E.P.A. 600/M4-82-020, 1982.

3. National Institute of Standards and Technology (NIST) National Voluntary Laboratory Accreditation Program (NVLAP) Program Handbook for Airborne Asbestos Analysis, NISTIR 89-4137, 1989.

APPENDIX C

Preparation and Use of Bulk Asbestos
Calibration Standards

C1.0 INTRODUCTION

Evaluation of the results from national proficiency testing programs for laboratories analyzing for asbestos in bulk materials indicates that laboratories have had, and continue to have, problems with quantitation of asbestos content, especially with samples having a low asbestos concentration.[1] For such samples, the mean value of asbestos content reported by laboratories may be four to ten times the true weight percent value. It is assumed that the majority of the laboratories quantify asbestos content by visual estimation, either stereomicroscopically or microscopically; therefore, the problem of quantitation must be attributed to lack of or inadequate calibration of microscopists.

As calibration standards for asbestos-containing bulk materials are not currently commercially available, laboratories should consider generating their own calibration materials. This may be done rather easily and inexpensively.

C2.0 MATERIALS AND APPARATUS

Relatively pure samples of asbestos minerals should be obtained. Chrysotile, amosite and crocidolite (SRM 1866) and anthophyllite, tremolite and actinolite (SRM 1867) are available from NIST. A variety of matrix materials are commercially available; included are calcium carbonate, perlite, vermiculite, mineral wool/fiberglass, and cellulose. Equipment, and materials needed to prepare calibration bulk materials are listed below.

- Analytical balance, readable to 0.001 gram
- Blender/mixer; multi-speed, ~ one quart capacity
- Filtration assembly, including vacuum flask, water aspirator and/or air pump (optional)
- HEPA-filtered hood with negative pressure
- Filters, 0.4μm pore size polycarbonate (optional)
- Beakers and assorted glassware, weigh boats, petri dishes, etc.
- Hot/warm plate

- Asbestos minerals
- Matrix materials
- Distilled water.

C3.0 MATERIAL FORMULATION PROCEDURES

The formulation procedure involves first weighing appropriate quantities of asbestos and matrix material to give the desired asbestos weight percent. The following formula may be used to determine the weights of asbestos and matrix materials needed to give a desired weight percent asbestos.

$$\frac{WTa}{Wa} = \frac{WTm}{Wm}$$

Where:

WTa = weight of asbestos in grams (to 0.001 gram)
WTm = weight of matrix materials in grams (to 0.001 gram)
Wa = weight percent asbestos
Wm = weight percent matrix

Example: The desired total weight for the calibration sample is – 10 grams containing 5% asbestos by weight. If 0.532 grams of asbestos are first weighed out, what corresponding weight of matrix material is required?

WTa = 0.532 grams
Wa = 5%
Wm = 95%

$$\frac{0.532}{5} = \frac{WTm}{95}$$

Then: WTm = 10.108 grams

The matrix is then placed into the pitcher of a standard over-the-counter blender, the pitcher being previously filled to approximately one-fourth capacity (8-10 ounces) with distilled water. Blending is performed at the lowest speed setting for approximately ten seconds which serves to disaggregate the matrix material. The asbestos is then added, with additional blending of approximately 30 seconds, again at the lowest speed setting. Caution should be taken not to overblend the asbestos-matrix mixture. This could result in a significant reduction in the size of the asbestos fibers causing a problem with detection at normal magnification during stereomicroscopic and microscopic analyses. Ingredients of the pitcher are then poured into a filtering apparatus, with thorough rinsing of the pitcher to ensure complete material removal. After filtering, the material is transferred to a foil dish which is placed on a hot plate. The material is covered and allowed to sit over low heat until drying is complete; intermittent stirring will speed the drying process. For fine-grained matrix materials such as gypsum, calcium carbonate, clays, etc., the sample is not filtered after the blending process. Instead, the ingredients in the pitcher are transferred into a series of shallow, glass (petri) dishes. The ingredients should be stirred well between each pouring to minimize the possible settling (and over-representation) of some components. The dishes are covered and placed on a hot plate until the contents are thoroughly dried. For small quantities of any matrix materials (15 grams or less), air-drying without prior filtering is generally very suitable for removing water from the prepared sample. For each material, the final step involves placing all formulated, dried subsamples into a plastic bag (or into one petri dish, for small quantities), where brief hand-mixing will provide additional blending and help to break up any clumps produced during drying. **All operations should be performed in a safety-hood with negative pressure.**

C4.0 ANALYSIS OF MATERIALS

All formulations should be examined with the stereomicroscope to determine homogeneity. Gravimetric analysis (ashing and/or acid dissolution) should be performed on those materials containing organic and/or acid-soluble components. Matrix materials to which no asbestos has been added should be analyzed by gravimetric analysis to determine the amount of nonashable or insoluble materials that are present. Several subsamples of each material should be analyzed by the gravimetric technique to provide information concerning the uniformity of the prepared materials. Experience has shown that the previously described formulation procedure results in relatively homogeneous materials.[2]

C4.1 Stereomicroscopic Analysis

Visual estimation of sample components using the stereomicroscope is in reality a comparison of the relative volumes of the components.[3] Therefore, differences in specific gravity between asbestos and matrix material must be considered and the relationship between weight percent and volume percent must be determined.[4] Materials such as expanded vermiculite, perlite, and cellulose have specific gravities significantly lower than asbestos minerals. Table C1 lists the specific gravities for the three most commonly encountered asbestos varieties and several common matrix materials.

TABLE C1. SPECIFIC GRAVITIES OF ASBESTOS VARIETIES AND MATRIX MATERIALS

Asbestos Type	Specific Gravity	Matrix Type	Specific Gravity
Chrysotile	2.6	Calcium Carbonate	2.7
		Gypsum	2.3
Amosite	3.2	Perlite	~0.4
		Vermiculite (expanded)	~0.3
		Mineral Wool	~2.5
Crocidolite	3.3	Fiberglass	~2.5
		Cellulose	~0.9

The conversion of weight percent asbestos to equivalent volume percent asbestos is given by the following formula:

$$\frac{\frac{Wa}{Ga}}{\frac{Wa}{Ga} + \frac{Wm}{Gm}} \times 100 = Va$$

where:

Wa	=	weight percent asbestos
Ga	=	specific gravity of asbestos
Wm	=	weight percent matrix
Gm	=	specific gravity of matrix
Va	=	volume percent asbestos

Example: Chrysotile and perlite have been combined to form a 5% asbestos calibration standard, by weight. What is the equivalent volume percent asbestos?

Wa = 5%
Ga = 2.6
Wm = 95%
Gm = 0.4

$$Va = \frac{\frac{5}{2.6}}{\frac{5}{2.6} + \frac{95}{0.4}} \times 100 = 0.8\%$$

Conversely, to convert volume percent asbestos to equivalent weight percent, the following formula may be used.

$$\frac{(Va)(Ga)}{(Va)(Ga)+(Vm)(Gm)} \times 100 = Wa$$

Vm = volume percent matrix

Example: A calibration standard consisting of amosite and cellulose is estimated to contain 2% asbestos, by volume. What is the equivalent weight percent asbestos?

Va = 2%
Ga = 3.2
Vm = 98%
Gm = 0.9

$$Wa = \frac{(2)(3.2)}{(2)(3.2)+(98)(0.9)} \times 100 = 6.77\%$$

Volume percentages should be calculated for all calibration materials prepared so that visual estimates determined by examination with the stereomicroscope may be compared to true volume concentrations.

Figure C1 illustrates the relationship between volume percent and weight percent of chrysotile mixed with vermiculite and cellulose respectively. It should be noted that when asbestos in a low weight percentage is mixed with matrix materials having low specific gravities (vermiculite, perlite), the resulting volume concentration of asbestos is very low. For example, a mixture containing three percent chrysotile by weight in a cellulose matrix would result in a volume percent asbestos of approximately 1.1%; in a vermiculite matrix, the resulting volume percent asbestos would be approximately 0.4%. In the latter case especially, an analyst might possibly fail to detect the asbestos or consider it to be present in only trace amounts.

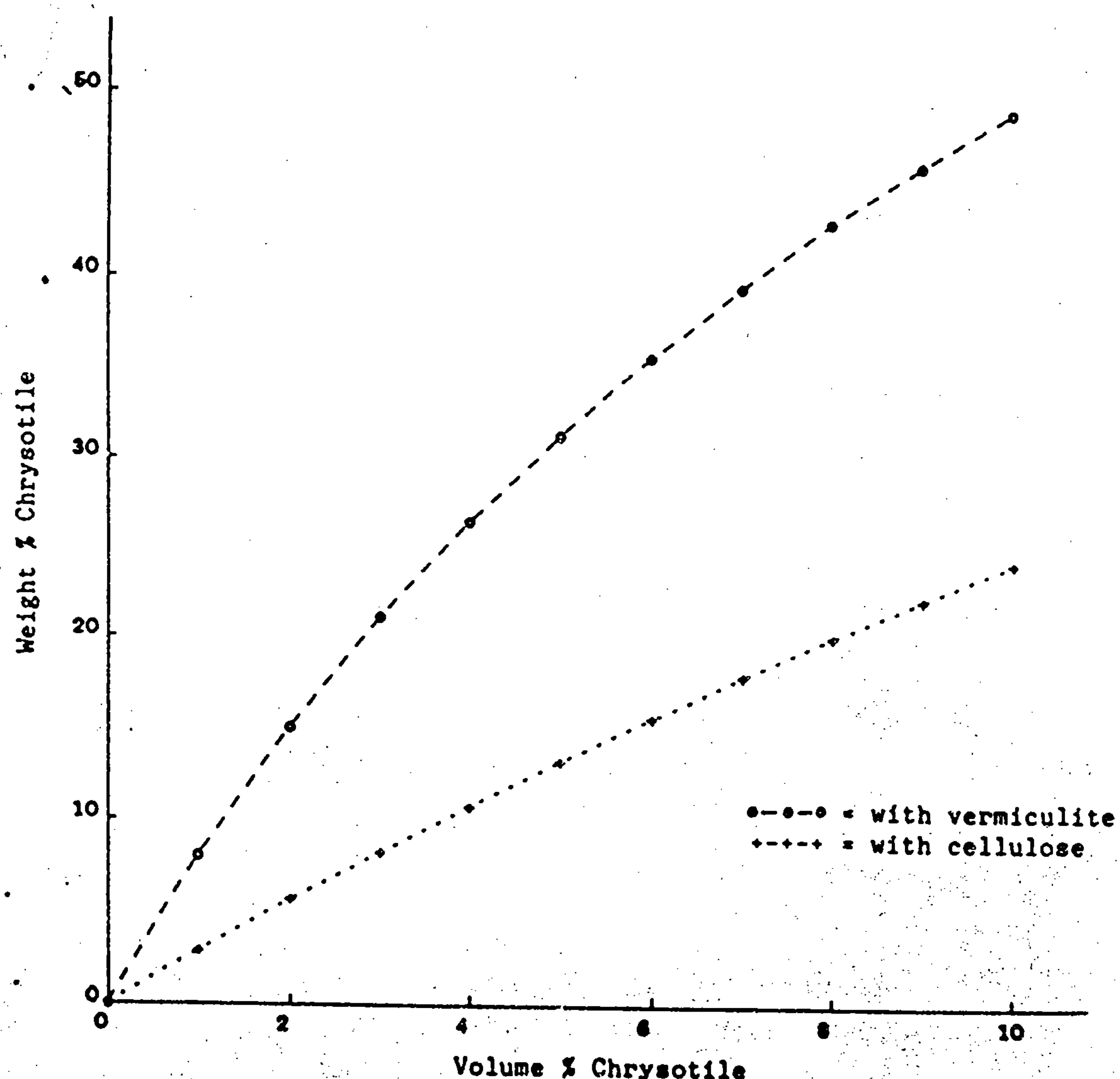


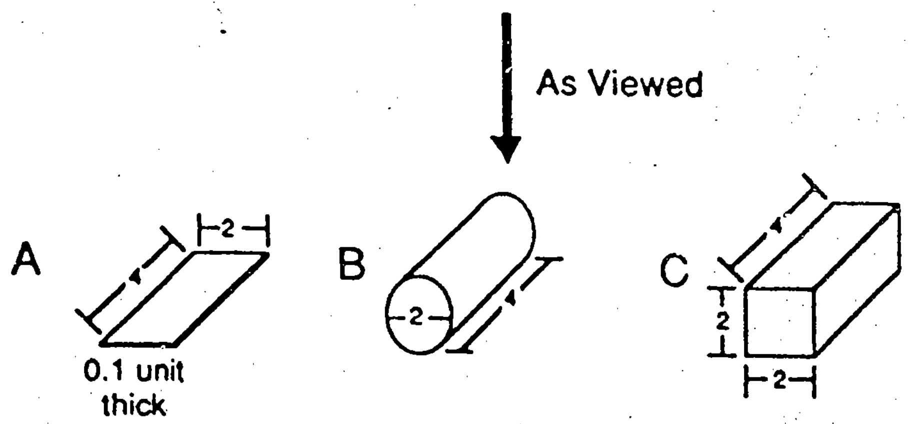
Figure C1. Relationship between volume % and weight % of chrysotile mixed with a)vermiculite and b) cellulose.

C4.2 Microscopical Analysis (PLM)

The polarized light microscope may be used to quantify asbestos and other components of a sample. Slide mounts are prepared from "pinch" samples of the calibration material and asbestos content is determined by visual area estimate and/or point counting. Both of these quantitation techniques are in fact estimates or measurements of the relative projected areas of particles as viewed in two dimensions on a microscope slide. For quantitation results to be meaningful, the following conditions should be met:

- The sample should be homogeneous for slide preparations, which are made from small pinches of the sample, to be representative of the total sample.
- Slide preparation should have an even distribution of particles and approach a one particle thickness (seldom achieved) to avoid particle overlap.
- All materials used should be identified and specific gravities determined in order to relate area percent to volume and/or weight percent.
- The size (thickness) relationship between matrix particles and asbestos fibers should be determined if the results based on projected area are to be related to volume and/or weight percent.

Particle characteristics can greatly affect the quantitation results obtained by visual area estimation or point counting. Figure C2 illustrates three hypothetical particle shapes of identical length and width (as viewed from above). Although the three-dimensional shape is different, the projected area is equal for all particles. The table accompanying Figure C2 presents data for each particle in terms of thickness, volume and projected area. It should be noted that although the projected areas may be equal, the volumes represented by the particles may vary by a factor of 20(0.8 vs 16 cubic units). It is obvious that quantitation of a sample consisting of a mixture of particles with widely ranging particle thicknesses could result in different results. For example, if a sample contained relatively thick bundles of asbestos and a fine-grained matrix such as clay or calcium carbonate, the true asbestos content (by volume) would likely be underestimated. Conversely, if a sample contained thick "books" of mica and thin bundles of asbestos, the asbestos content (by volume) would likely be overestimated.



Particle	Thickness	Volume	Projected Area
A	0.1 units	0.8 cubic units	8 sq. units
B	2 units	12.6 cubic units	8 sq. units
C	2 units	16 cubic units	8 sq. units

Note that although all particles have the same projected area, particle C volume is 20x that of particle A.

Figure C2. Relationship of projected area to volume and thickness for three different particles as viewed on a slide mount.

Table C2 illustrates several examples of expected results from area estimates or point counting of samples in which the asbestos fibers and matrix particles differ in thickness.

TABLE C2. RELATIONSHIP OF WEIGHT PERCENT, VOLUME PERCENT AND PARTICLE THICKNESS TO QUANTITATION RESULTS

Composition of Sample In Wt. %	Theoretical Vol. % Asbestos	Thickness Factor* (Matrix/Asbestos)	Expected Area %
1% Amosite 99% Calcium Carbonate	0.9	0.5	0.4
1% Amosite 99% Calcium Carbonate	0.9	1	0.9
1% Amosite 99% Calcium Carbonate	0.9	2	1.8
1% Amosite 99% Vermiculite	0.1	1	0.1
1% Amosite 99% Vermiculite	0.1	10	1.0
1% Amosite 99% Vermiculite	0.1	20	2.0
1% Amosite 99% Vermiculite	0.1	30	2.9

* Value represents the relationship between the mean thickness of the matrix particles compared to the mean thickness of the asbestos particles.

It should be noted that it is not uncommon for matrix particle thickness to differ greatly from asbestos fiber thickness, especially with matrix materials such as vermiculite and perlite; vermiculite and perlite particles may be 20 - 30 times as thick as the asbestos fibers. The general size relationships between matrix particles and asbestos fibers may be determined by scanning slide mounts of a sample. A micrometer ocular enables the microscopist to actually measure particle sizes.

If a thickness factor can be determined for a calibration sample of known volume proportions of asbestos and matrix materials, an expected equivalent projected area asbestos can be calculated using the following formula:

$$\frac{Va}{\frac{Vm}{T} + Va} \times 100 = Aa$$

where:

Va	=	true volume percent asbestos
Vm	=	true volume percent matrix
T	=	thickness factor (mean size matrix particle/mean size asbestos fiber)
Aa	=	expected projected area percent asbestos

Example: A calibration standard of known weight percent asbestos is determined, by factoring in component specific gravities, to be 5.0% asbestos by volume. The matrix particles are estimated to be ten times thicker than the asbestos fibers. What would be the expected projected area percentage of asbestos?

$Va = 5\%$
$Vm = 95\%$
$T = 10$

$$Aa = \frac{5}{\frac{95}{10} + 5} \times 100 = 34.5\%$$

Conversely, to convert projected area percent asbestos to equivalent volume percent, the following formula may be used:

$$\frac{Aa}{T(Am) + Aa} \times 100 = Va$$

Where: Am = projected area matrix

Example: A slide containing a subsample of an amosite/mineral wool calibration standard is determined by point counting to have a projected area asbestos of 18.6%. If the mineral wool fibers are estimated to be six times the asbestos fibers, in diameter, what is the equivalent volume percent asbestos?

$$Am = 81.4\%$$
$$Aa = 18.6\% \qquad Va = \frac{(18.6)}{6(81.4) + 18.6} \times 100 = 3.67\%$$
$$T = 6$$

Based on specific gravity values listed in Table 1C and on the above volume asbestos determination, what is the equivalent weight percent asbestos in the sample?

$$Va = 3.67\%$$
$$Ga = 3.2 \qquad Wa = \frac{(3.67)(3.2)}{(3.67)(3.2) + (96.33)(2.5)} \times 100 = 4.7\%$$
$$Vm = 96.33\%$$
$$Gm = 2.5$$

C5.0 USE OF CALIBRATION STANDARDS FOR QA/QC

Once the materials have been formulated and thoroughly characterized by all techniques to determine their suitability as calibration standards, a system for incorporating them into the QA/QC program should be established. Someone should be designated (QA officer, lab supervisor, etc.) to control the distribution of standards and to monitor the analysis results of the microscopists. Both precision and accuracy may be monitored with the use of suitable standard sets.

Records such as range charts, control charts, etc. may be maintained for volume (stereomicroscopic estimates), area (PLM) estimates and point counts. For point counts and area estimates, relatively permanent slides may be made using epoxy or Melt Mount ®. Such slides may be very accurately quantified over time as to point count values, and due to their very long shelf life, may be used for QA/QC purposes almost indefinitely.

C6.0 REFERENCES

1. "Analysis Summaries for Samples used in NIST Proficiency Testing", National Institute of Standards and Technology (NIST) National Voluntary Laboratory Accreditation Program (NVLAP) for Bulk Asbestos, January 1989 to present.

2. Harvey, B. W., R. L. Perkins, J. G. Nickerson, A. J. Newland and M. E. Beard, "Formulating Bulk Asbestos Standards", **Asbestos Issues**, April 1991.

3. Perkins, R. L. and M. E. Beard, "Estimating Asbestos Content of Bulk Materials", **National Asbestos Council Journal**, Vol. 9, No. 1, 1991, pp. 27-31.

4. **Asbestos Content in Bulk Insulation Samples: Visual Estimates and Weight Composition**, U.S. Environmental Protection Agency 560/5-88-011, 1988.

APPENDIX D

Special-Case Building Materials

Asbestos laboratories are now called upon to analyze many types of bulk building materials that are very difficult to characterize by routine PLM analysis. These materials are dominantly nonfriable and can be grouped into the following categories:

- Cementitious Products (pipe, sheeting, etc.)
- Viscous Matrix Products (adhesives, cements, coatings, etc.)
- Vinyl Materials (vinyl floor tile, sheeting)
- Asphaltic Roofing Materials (shingles, roll roofing)
- Miscellaneous Products (paints, coatings, friction plates, gaskets, etc.)

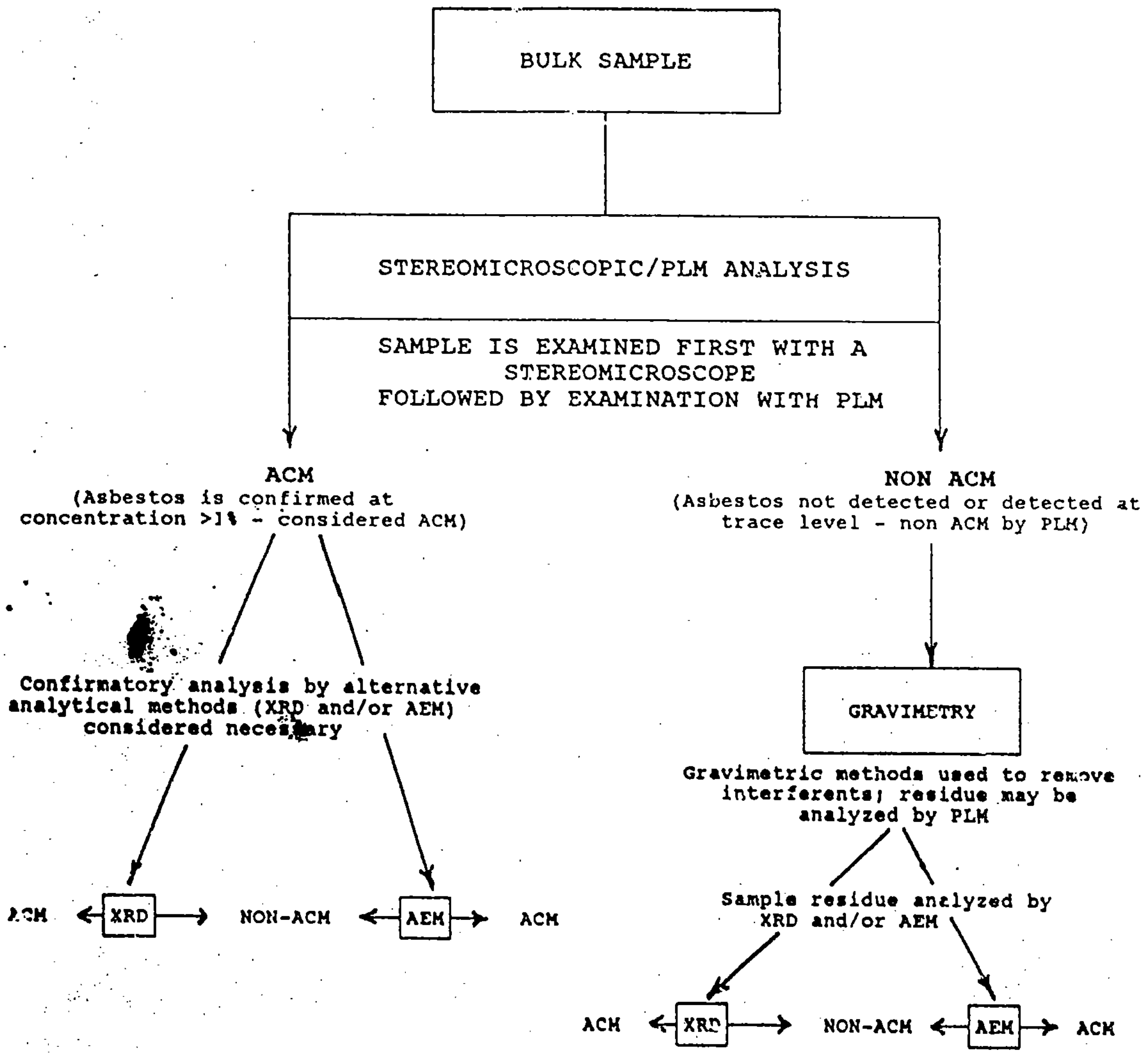
Materials characterized by interfering binder/matrix, low asbestos content, and/or small fiber size may require that additional sample treatment(s) and analysis be performed beyond routine PLM analysis. The sample treatment(s) required is(are) determined by the dominant nonasbestos sample components (see Section 2.3, Gravimetry). Materials containing an appreciable amount of calcareous material may be treated by dissolution with hydrochloric acid. Samples containing organic binders such as vinyl, plasticizers, esters, asphalts, etc. can be treated with organic solvents or ashed in a muffle furnace (preferred method) or low temperature plasma asher to remove unwanted components. Materials containing cellulose, synthetic organic fibers, textiles, etc. may also be ashed in a muffle furnace or low temperature plasma asher.

The method chosen for analysis of a sample after treatment is dependent on asbestos concentration and/or fiber size. An examination of the sample residue by PLM may disclose asbestos if the fibers are large enough to be resolved by the microscope, but additional analytical methods are required if the sample appears negative. Analysis by XRD is not fiber-size dependent, but may be limited by low concentration of asbestos and the presence of interfering mineral phases. In addition, the XRD method does not differentiate between fibrous and nonfibrous varieties of a mineral. Analysis by AEM is capable of providing positive identification of asbestos type(s) and semi-quantitation of asbestos content.

The following flowchart illustrates a possible scheme for the analysis of special-case building materials.

NOTE: Preliminary studies indicate that the XRD method is capable of detecting serpentine (chrysotile) in floor tile samples without extensive sample preparation prior to XRD analysis. XRD analysis of small, intact sections of floor tile yielded diffraction patterns that confirmed the presence of serpentine, even at concentrations of ~one percent by weight. TEM analysis of these same tiles confirmed the presence of chrysotile asbestos. With further investigation, this method may prove applicable to other types of nonfriable materials.

FLOWCHART FOR QUALITATIVE ANALYSIS OF SPECIAL CASE BUILDING MATERIALS SUCH AS FLOOR TILES, ASPHALTIC MATERIALS, VISCOUS MATRIX MATERIALS, ETC.*



*Although this flowchart is applicable to all bulk materials, it is primarily intended to be used with known problem materials that are difficult to analyze by PLM due to low asbestos concentration, and/or small fiber size, and/or interfering binder/matrix. In addition to being qualitative, the results may also be semi-quantitative. It should not be assumed that all samples need to be analyzed by AEM and XRD. The flowchart simply illustrates options for methods of analysis. Alternate methods such as SEM may be applicable to some bulk materials.

*U.S. GOVERNMENT PRINTING OFFICE 1993 -750 -002/80237

TETRA TECH

APPENDIX H:
ASTM D5755-03

Designation: D 5755 – 03

Standard Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Structure Number Surface Loading[1]

This standard is issued under the fixed designation D 5755; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers a procedure to (*a*) identify asbestos in dust and (*b*) provide an estimate of the surface loading of asbestos in the sampled dust reported as the number of asbestos structures per unit area of sampled surface.

1.1.1 If an estimate of the asbestos mass is to be determined, the user is referred to Test Method D 5756.

1.2 This test method describes the equipment and procedures necessary for sampling, by a microvacuum technique, non-airborne dust for levels of asbestos structures. The non-airborne sample is collected inside a standard filter membrane cassette from the sampling of a surface area for dust which may contain asbestos.

1.2.1 This procedure uses a microvacuuming sampling technique. The collection efficiency of this technique is unknown and will vary among substrates. Properties influencing collection efficiency include surface texture, adhesiveness, electrostatic properties and other factors.

1.3 Asbestos identified by transmission electron microscopy (TEM) is based on morphology, selected area electron diffraction (SAED), and energy dispersive X-ray analysis (EDXA). Some information about structure size is also determined.

1.4 This test method is generally applicable for an estimate of the surface loading of asbestos structures starting from approximately 1000 asbestos structures per square centimetre.

1.4.1 The procedure outlined in this test method employs an indirect sample preparation technique. It is intended to disperse aggregated asbestos into fundamental fibrils, fiber bundles, clusters, or matrices that can be more accurately quantified by transmission electron microscopy. However, as with all indirect sample preparation techniques, the asbestos observed for quantification may not represent the physical form of the asbestos as sampled. More specifically, the procedure described neither creates nor destroys asbestos, but it may alter the physical form of the mineral fibers.

1.5 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water[2]

D 3195 Practice for Rotameter Calibration[3]

D 3670 Guide for Determination of Precision and Bias of Methods of Committee D22[3]

D 5756 Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Mass Surface Loading[3]

D 6620 Practice for Determining a Detection Limit for Asbestos Measurements Based on Counts[3]

3. Terminology

3.1 *Definitions:*

3.1.1 *asbestiform*—a special type of fibrous habit in which the fibers are separable into thinner fibers and ultimately into fibrils. This habit accounts for greater flexibility and higher tensile strength than other habits of the same mineral. For more information on asbestiform mineralogy, see Refs **(1)**,[4] **(2)** and **(3)**.

3.1.2 *asbestos*—a collective term that describes a group of naturally occurring, inorganic, highly fibrous, silicate dominated minerals, which are easily separated into long, thin, flexible fibers when crushed or processed.

[1] This test method is under the jurisdiction of ASTM Committee D22 on Sampling and Analysis of Atmospheres and is the direct responsibility of Subcommittee D22.07 on Sampling and Analysis of Asbestos.

Current edition approved April 10, 2003. Published June 2003. Originally approved in 1995. Last previous edition approved in 2002 as D 5755 - 02.

[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] *Annual Book of ASTM Standards*, Vol 11.03.

[4] The boldface numbers in parentheses refer to the list of references at the end of this test method.

3.1.2.1 *Discussion*—Included in the definition are the asbestiform varieties of: serpentine (chrysotile); riebeckite (crocidolite); grunerite (grunerite asbestos); anthophyllite (anthophyllite asbestos); tremolite (tremolite asbestos); and actinolite (actinolite asbestos). The amphibole mineral compositions are defined according to nomenclature of the International Mineralogical Association **(3)**.

Asbestos	Chemical Abstract Service No.[5]
Chrysotile	12001-29-5
Crocidolite	12001-28-4
Grunerite Asbestos	12172-73-5
Anthophyllite Asbestos	77536-67-5
Tremolite Asbestos	77536-68-6
Actinolite Asbestos	77536-66-4

3.1.3 *fibril*—a single fiber that cannot be separated into smaller components without losing its fibrous properties or appearance.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *aspect ratio*—the ratio of the length of a fibrous particle to its average width.

3.2.2 *bundle*—a structure composed of three or more fibers in a parallel arrangement with the fibers closer than one fiber diameter to each other.

3.2.3 *cluster*—a structure with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated from the group; groupings of fibers must have more than two points touching.

3.2.4 *debris*—materials that are of an amount and size (particles greater than 1 mm in diameter) that can be visually identified as to their source.

3.2.5 *dust*—any material composed of particles in a size range of <1 mm.

3.2.6 *fiber*—a structure having a minimum length of 0.5 μm, an aspect ratio of 5:1 or greater, and substantially parallel sides **(4)**.

3.2.7 *fibrous*—of a mineral composed of parallel, radiating, or interlaced aggregates of fibers, from which the fibers are sometimes separable. That is, the crystalline aggregate may be referred to as fibrous even if it is not composed of separable fibers, but has that distinct appearance. The term fibrous is used in a general mineralogical way to describe aggregates of grains that crystallize in a needle-like habit and appear to be composed of fibers. Fibrous has a much more general meaning than asbestos. While it is correct that all asbestos minerals are fibrous, not all minerals having fibrous habits are asbestos.

3.2.8 *indirect preparation*—a method in which a sample passes through one or more intermediate steps prior to final filtration.

3.2.9 *matrix*—a structure in which one or more fibers, or fiber bundles that are touching, are attached to, or partially concealed by a single particle or connected group of nonfibrous particles. The exposed fiber must meet the fiber definition (see 3.2.6).

3.2.10 *structures*—a term that is used to categorize all the types of asbestos particles which are recorded during the analysis (such as fibers, bundles, clusters, and matrices). Final results of the test are always expressed in asbestos structures per square centimetre.

4. Summary of Test Method

4.1 The sample is collected by vacuuming a known surface area with a standard 25 or 37 mm air sampling cassette using a plastic tube that is attached to the inlet orifice which acts as a nozzle. The sample is transferred from inside the cassette to an aqueous suspension of known volume. Aliquots of the suspension are then filtered through a membrane. A section of the membrane is prepared and transferred to a TEM grid using the direct transfer method. The asbestiform structures are identified, sized, and counted by TEM, using SAED and EDXA at a magnification of 15 000 to 20 000X.

5. Significance and Use

5.1 This microvacuum sampling and indirect analysis method is used for the general testing of non-airborne dust samples for asbestos. It is used to assist in the evaluation of dust that may be found on surfaces in buildings such as ceiling tiles, shelving, electrical components, duct work, carpet, etc. This test method provides an index of the surface loading of asbestos structures in the dust per unit area analyzed as derived from a quantitative TEM analysis.

5.1.1 This test method does not describe procedures or techniques required to evaluate the safety or habitability of buildings with asbestos-containing materials, or compliance with federal, state, or local regulations or statutes. It is the user's responsibility to make these determinations.

5.1.2 At present, no relationship has been established between asbestos-containing dust as measured by this test method and potential human exposure to airborne asbestos. Accordingly, the users should consider other available information in their interpretation of the data obtained from this test method.

5.2 This definition of dust accepts all particles small enough to pass through a 1 mm (No. 18) screen. Thus, a single, large asbestos containing particle(s) (from the large end of the particle size distribution) dispersed during sample preparation may result in anomalously large asbestos surface loading results in the TEM analyses of that sample. It is, therefore, recommended that multiple independent samples are secured from the same area, and that a minimum of three samples be analyzed by the entire procedure.

6. Interferences

6.1 The following minerals have properties (that is, chemical or crystalline structure) which are very similar to asbestos minerals and may interfere with the analysis by causing a false positive to be recorded during the test. Therefore, literature references for these materials must be maintained in the laboratory for comparison to asbestos minerals so that they are not misidentified as asbestos minerals.

6.1.1 *Antigorite.*

6.1.2 *Palygorskite (Attapulgite).*

6.1.3 *Halloysite.*

6.1.4 *Pyroxenes.*

6.1.5 *Sepiolite.*

6.1.6 *Vermiculite scrolls.*

[5] The non-asbestiform variations of the minerals indicated in 3.1.3 have different Chemical Abstract Service (CAS) numbers.

6.1.7 *Fibrous talc.*

6.1.8 Hornblende and other amphiboles other than those listed in 3.1.2.

6.2 Collecting any dust particles greater than 1 mm in size in this test method may cause an interference and, therefore, must be avoided.

7. Materials and Equipment

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.[6]

7.2 *Transmission Electron Microscope (TEM)*, an 80 to 120 kV TEM, capable of performing electron diffraction, with a fluorescent screen inscribed with calibrated gradations, is required. The TEM must be equipped with energy dispersive X-ray spectroscopy (EDXA) and it must have a scanning transmission electron microscopy (STEM) attachment or be capable of producing a spot size of less than 250 nm in diameter in crossover.

7.3 *Energy Dispersive X-ray System (EDXA).*

7.4 *High Vacuum Carbon Evaporator*, with rotating stage.

7.5 *High Efficiency Particulate Air (HEPA)*, filtered negative flow hood.

7.6 *Exhaust or Fume Hood.*

7.7 *Particle-free Water* (ASTM Type II, see Specification D 1193).

7.8 *Glass Beakers* (50 mL).

7.9 *Glass Sample Containers*, with wide mouth screw cap (200 mL) or equivalent sealable container (height of the glass sample container should be approximately 13 cm high by 6 cm wide).

7.10 *Waterproof Markers.*

7.11 *Forceps* (tweezers).

7.12 *Ultrasonic Bath*, table top model (100 W).

7.13 *Graduated Pipettes* (1, 5, 10 mL sizes), glass or plastic.

7.14 *Filter Funnel*, either 25 mm or 47 mm, glass or disposable. Filter funnel assemblies, either glass or disposable plastic, and using either a 25 mm or 47 mm diameter filter.

7.15 *Side Arm Filter Flask*, 1000 mL.

7.16 *Mixed Cellulose Ester (MCE) Membrane Filters*, 25 or 47 mm diameter, $\leq$0.22 µm and 5 µm pore size.

7.17 *Polycarbonate (PC) Filters*, 25 or 47 mm diameter, $\leq$0.2 µm pore size.

7.18 *Storage Containers*, for the 25 or 47 mm filters (for archiving).

7.19 *Glass Slides*, approximately 76 by 25 mm in size.

7.20 *Scalpel Blades*, No. 10, or equivalent.

7.21 *Cabinet-type Desiccator*, or low temperature drying oven.

7.22 *Chloroform*, reagent grade.

7.23 *Acetone*, reagent grade.

7.24 *Dimethylformamide (DMF).*

7.25 *Glacial Acetic Acid.*

7.26 *1-methyl-2-pyrrolidone.*

7.27 *Plasma Asher*, low temperature.

7.28 *pH Paper.*

7.29 *Air Sampling Pump*, low volume personal-type, capable of achieving a flow rate of 1 to 5 L/min.

7.30 *Rotameter.*

7.31 *Air Sampling Cassettes*, 25 mm or 37 mm, containing 0.8µ m or smaller pore size MCE or PC filters.

7.32 *Cork Borer*, 7 mm.

7.33 *Non-Asbestos Mineral*, references as outlined in 6.1.

7.34 *Asbestos Standards*, as outlined in 3.1.2.

7.35 *Tygon[7] Tubing*, or equivalent.

7.36 *Small Vacuum Pump*, that can maintain a pressure of 92 kPa.

7.37 *Petri Dishes*, large glass, approximately 90 mm in diameter.

7.38 *Jaffe Washer*, stainless steel or aluminum mesh screen, 30 to 40 mesh, and approximately 75 mm by 50 mm in size.

7.39 *Copper TEM Finder Grids*, 200 mesh.

7.40 *Carbon Evaporator Rods.*

7.41 *Lens Tissue.*

7.42 *Ashless Filter Paper Filters*, 90 mm diameter.

7.43 *Gummed Paper Reinforcement Rings.*

7.44 *Wash Bottles*, plastic.

7.45 *Reagent Alcohol*, HPLC Grade (Fisher A995 or equivalent).

7.46 *Opening Mesh Screen*, plastic, 1.0 by 1.0 mm, (Spectra-Mesh #146410 or equivalent).

7.47 *Diffraction Grating Replica.*

8. Sampling Procedure for Microvacuum Technique

8.1 For sampling asbestos-containing dust in either indoor or outdoor environments, commercially available cassettes must be used. Air monitoring cassettes containing 25 mm or 37 mm diameter mixed cellulose ester (MCE) or polycarbonate (PC) filter membranes with a pore size less than or equal to 0.8 µm are required (7.31). The number of samples collected depends upon the specific circumstances of the study.

8.2 Maintain a log of all pertinent sampling information and sampling locations.

8.3 Sampling pumps and flow indicators shall be calibrated using a certified standard apparatus or assembly (see Practice D 3195 and 7.29).

8.4 Record all calibration information **(5)**.

8.5 Perform a leak check of the sampling system at each sampling site by activating the pump (7.29) with the closed sampling cassette in line. Any air flow shows that a leak is present that must be eliminated before initiating the sampling operation.

[6] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

[7] Tygon is a registered trademark of the DuPont Co.

8.6 Attach the sampling cassette to the sampling pump at the outlet side of the cassette with plastic tubing (7.35). The plastic tubing must be long enough in that the sample areas can be reached without interference from the sampling pump. Attach a clean, approximately 25.4 mm long piece of plastic tubing (6.35 mm internal diameter) directly to the inlet orifice. Use this piece of tubing as the sampling nozzle. Cut the sampling end of the tubing at a 45° angle as illustrated in Fig. 1. The exact design of the nozzle is not critical as long as some vacuum break is provided to avoid simply pushing the dust around on the surface with the nozzle rather than vacuuming it into the cassette. The internal diameter of the nozzle and flow rate of the pump may vary as long as the air velocity is 100 (±10) cm/s. This air velocity calculation is based on an internal sampling tube diameter of 6.35 mm at a flow rate of 2 L/min.

8.7 Measure and determine the sample area of interest. A sample area of 100 cm^2 is vacuumed until there is no visible dust or particulates matter remaining. Perform a minimum of two orthogonal passes on the surface within a minimum of 2 min of sampling time. Avoid scraping or abrading the surface being sampled. (Do not sample any debris or dust particles greater than 1 mm in diameter (see 4.2).) Smaller or larger areas can be sampled, if needed. For example, some surfaces of interest may have a smaller area than 100 cm^2. Less dusty surfaces may require vacuuming of larger areas. Unlike air samples, the overloading of the cassettes with dust will not be a problem. As defined in 3.2.5, only dust shall be collected for this analysis.

8.8 At the end of sample collection, invert the cassette so that the nozzle inlet faces up before shutting off the power to the pump. The nozzle is then sealed with a cassette end-plug and the cassette/nozzle taped or appropriately packaged to prevent separation of the nozzle and cassette assembly. A second option is the removal of the nozzle from the cassette, then plugging of the cassette and shipment of the nozzle (also plugged at both ends) sealed in a separate closeable plastic bag. A third option is placing the nozzle inside the cassette for shipment. The nozzle is always saved and rinsed because a significant percentage of the dust drawn from a lightly loaded surface may adhere to the inside walls of the tubing.

8.9 Check that all samples are clearly labeled, that all dust sampling information sheets are completed, and that all pertinent information has been enclosed, in accordance with laboratory quality control practices, before transfer of the samples to the laboratory. Include an unused cassette and nozzle as a field blank.

8.10 Wipe off the exterior surface of the cassettes with disposable wet towels (baby wipes) prior to packaging for shipment.

9. Sample Shipment

9.1 Ship dust samples to an analytical laboratory in a sealed container, but separate from any bulk or air samples. The cassettes must be tightly sealed and packed in a material free of fibers or dust to minimize the potential for contamination. Plastic "bubble pack" is probably the most appropriate material for this purpose.

10. Sample Preparation

10.1 Under a negative flow HEPA hood (7.5), carefully wet-wipe the exterior of the cassettes to remove any possible contamination before taking cassettes into a clean preparation area.

10.2 Perform sample preparation in a clean facility that has a separate work area from both the bulk and air sample preparation areas.

10.3 Initial specimen preparation shall take place in a clean HEPA filtered negative pressure hood to avoid any possible contamination of the laboratory or personnel, or both, by the potentially large number of asbestos structures in an asbestos-containing dust sample. Cleanliness of the preparation area hoods is measured by the cumulative process blank surface loadings (see Section 11).

10.4 All sample preparation steps 10.4.1-10.4.6 shall take place in the dust preparation area inside a HEPA hood.

10.4.1 Remove the upper plug from the sample cassette and carefully introduce approximately 10 mL solution of a 50/50 mixture of particle-free water and reagent alcohol into the cassette using a plastic wash bottle (7.44). If the plugged nozzle was left attached to the cassette, then remove the plug and introduce the water/alcohol solution into the cassette through the tubing, and then remove the tubing, if it is visibly clean.

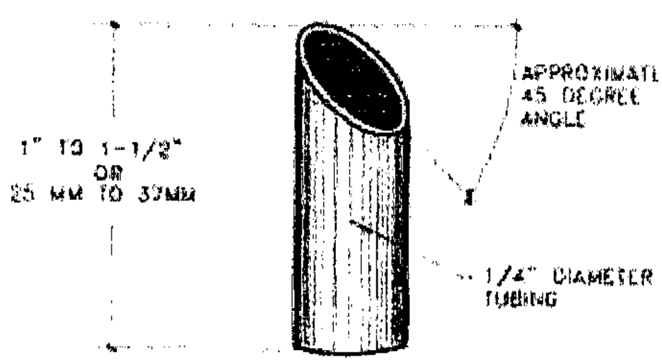


FIG. 1 Example of the Tubing Nozzle

10.4.2 Replace the upper plug or the sample cap and lightly shake the dust solution by hand for 3 s.

10.4.3 Remove the entire cap of the cassette and pour the suspension through a 1.0 by 1.0 mm opening screen (7.46) into a pre-cleaned 200 mL glass specimen bottle (7.9). All visible traces of the sample contained in the cassette shall be rinsed through the screen into the specimen bottle with a plastic wash bottle containing the 50/50 solution of particle-free water and alcohol. Repeat this procedure two additional times for a total of three washings. Next, rinse the nozzle two or three times through the screen into the specimen bottle with the 50/50 mixture of water and alcohol. Typically, the total amount of the 50/50 mixture used in the rinse is 50 to 75 mL. Discard the 1.0 by 1.0 mm screen and bring the volume of suspension in the specimen bottle up to the 100 mL mark on the side of the bottle with particle-free water only.

10.4.4 Adjust the pH of the suspension to 3 to 4 using a 10.0 % solution of acetic acid. Use pH paper for testing. Filter the suspension within 24 h to avoid problems associated with bacterial and fungal growth.

10.4.5 Use either a disposable plastic filtration unit or a glass filtering unit (7.14) for filtration of aliquots of the suspension. The ability of an individual filtration unit to produce a uniform distribution may be tested by the filtration of a colored particulate solution such as diluted India ink (solution of carbon black).

10.4.5.1 If a disposable plastic filtration unit is used, then unwrap a new disposable plastic filter funnel unit (either 25 or 47 mm diameter) and remove the tape around the base of the funnel. Remove the funnel and discard the top filter supplied with the apparatus, retaining the coarse polypropylene support pad in place. Assemble the unit with the adapter and a properly sized neoprene stopper, and attach the funnel to the 1000 mL side-arm vacuum flask (7.15). Place a 5.0 μm pore size MCE (backing filter) on the support pad. Wet it with a few mL of particle-free water and place an MCE (7.16) or PC filter (≤0.22 μm pore size) (7.17) on top of the backing filter. Apply a vacuum (7.36), ensuring that the filters are centered and pulled flat without air bubbles. Any irregularities on the filter surface requires the discard of that filter. After the filter has been seated properly, replace the funnel and reseal it with the tape. Return the flask to atmospheric pressure.

10.4.5.2 If a glass filtration unit is used, place a 5 μm pore size MCE (backing filter) on the glass frit surface. Wet the filter with particle-free water, and place an MCE or PC filter (≤0.22 μm pore size) on top of the backing filter. Apply a vacuum, ensuring that the filters are centered and pulled flat without air bubbles. Replace the filters if any irregularities are seen on the filter surface. Before filtration of each set of sample aliquots, prepare a blank filter by filtration of 50 mL of particle-free water. If aliquots of the same sample are filtered in order of increasing surface loading, the glass filtration unit need not be washed between filtration. After completion of the filtration, do not allow the filtration funnel assembly to dry because contamination is then more difficult to remove. Wash any residual solution from the filtration assembly by holding it under a flow of water, then rub the surface with a clean paper towel soaked in a detergent solution. Repeat the cleaning operation, and then rinse two times in particle-free water.

10.4.6 With the flask at atmospheric pressure, add 20 mL of particle-free water into the funnel. Cover the filter funnel with its plastic cover if the disposable filtering unit is used.

10.4.7 Briefly hand shake (3 s) the capped bottle with the sample suspension, then place it in a tabletop ultrasonic bath (7.12) and sonicate for 3.0 min. Maintain the water level in the sonicator at the same height as the suspension in sample bottle. The ultrasonic bath shall be calibrated as described in 20.5. The ultrasonic bath must be operated at equilibrium temperature. After sonicating, return the sample bottle to the work surface of the HEPA hood. Preparation steps 10.4.8-10.4.14 shall be carried out in this hood.

10.4.8 Shake the suspension lightly by hand for 3 s, then let it rest for 2.0 min to allow large particles to settle to the bottom of the bottle or float to the surface.

10.4.9 Estimate the amount of liquid to be withdrawn to produce an adequate filter preparation. Experience has shown that a light staining of the filter surface will yield a suitable preparation for analysis. Filter at least 1.0 mL, but no more than half the total volume. If after examination in the TEM, the smallest volume measured (1.0 mL) (7.13) yields an overloaded sample, then perform additional serial dilutions of the suspension. If it is estimated that less than 1.0 mL of suspension has to be filtered because of the density of the suspension, perform a serial dilution.

10.4.9.1 If serial dilutions are required, repeat step 10.4.8 before the serial dilution portion is taken. Do not re-sonicate the original suspension or any serial dilutions. The recommended procedure for a serial dilution is to mix 10 mL of the sample suspension with 90 mL of particle-free water in a clean sample bottle to obtain a 1:10 serial dilution. Follow good laboratory practices when performing dilutions.

10.4.10 Insert a new disposable pipette halfway into the sample suspension and withdraw a portion. Avoid pipetting any of the large floating or settled particles. Uncover the filter funnel and dispense the mixture from the pipette into the water in the funnel.

10.4.11 Apply vacuum to the flask and draw the mixture through the filter.

10.4.12 Discard the pipette.

10.4.13 Disassemble the filtering unit and carefully remove the sample filter with fine tweezers (7.11). Place the completed sample filter particle side up, into a precleaned, labeled, disposable, plastic petri dish (7.48) or other similar container.

10.4.14 In order to ensure that an optimally-loaded filter is obtained, it is recommended that filters be prepared from several different aliquots of the dust suspension. For this series of filters, it is recommended that the volume of each aliquot of the original suspension be a factor of five higher than the previous one. If the filters are prepared in order of increasing aliquot volume, all of the filters for one sample can be prepared using one plastic disposable filtration unit, or without cleaning of glass filtration equipment between individual filtration. Before withdrawal of each aliquot from the sample, shake the suspension without additional sonification and allow to rest for 2 min.

10.4.15 There are many practical methods for drying MCE filters. The following are two examples that can be used: (*1*) dry MCE filters for at least 12 h (over desiccant) in an airtight cabinet-type desiccator (7.21); (*2*) to shorten the drying time (if desired), remove a plug of the damp filter and attach it to a glass slide (7.19) as described in 12.1.2 and 12.1.3. Place the slide with a filter plug or filter plugs (up to eight plugs can be attached to one slide) on a bed of desiccant, in the desiccator for 1 h.

10.4.16 PC filters do not require lengthy drying before preparation, but shall be placed in a desiccator for at least 30 min before preparation.

10.5 Prepare TEM specimens from small sections of each dried filter using the appropriate direct transfer preparation method.

11. Blanks

11.1 Prepare sample blanks that include both a process blank (50 mL of particle-free water) for each set of samples analyzed and one unused filter from each new box of sample filters (MCE or PC) used in the laboratory. If glass filtering units are used, prepare and analyze a process blank each time the filtering unit is cleaned. Blanks will be considered contaminated, if after analysis, they are shown to contain more than 53 asbestos structures per square millimetre. This generally corresponds to three or four asbestos structures found in ten grid openings. The source of the contamination must be found before any further analysis can be performed. Reject samples that were processed along with the contaminated blanks and prepare new samples after the source of the contamination is found.

11.2 Prepare field blanks which are included with sample sets in the same manner as the samples, to test for contamination during the sampling, shipping, handling, and preparation steps of the method.

12. TEM Specimen Preparation of Mixed Cellulose Ester (MCE) Filters

NOTE 1—Use of either the acetone or the diamethylformamide-acetic acid method is acceptable.

12.1 *Acetone Fusing Method*:

12.1.1 Remove a section (a plug) from any quadrant of the sample and blank filters. Sections can be removed from the filters using a 7 mm cork borer (7.32). The cork borer must be wet wiped after each time a section is removed.

12.1.2 Place the filter section (particle side up) on a clean microscope slide. Affix the filter section to the slide with a gummed page reinforcement (7.43), or other suitable means. Label the slide with a glass scribing tool or permanent marker (7.10).

12.1.3 Prepare a fusing dish from a glass petri dish (7.37) and a metal screen bridge (7.38) with a pad of five to six ashless paper filters (7.42) and place in the bottom of the petri dish (4). Place the screen bridge on top of the pad and saturate the filter pads with acetone. Place the slide on top of the bridge in the petri dish and cover the dish. Wait approximately 5 min for the sample filter to fuse and clear.

12.2 *Dimethylformamide-Acetic Acid Method*:

12.2.1 Place a drop of clearing solution that consists of 35 % dimethylformamide (DMF), 15 % glacial acetic acid, and 50 % Type II water (v/v) on a clean microscope slide. Gauge the amount used so that the clearing solution just saturates the filter section.

12.2.2 Carefully lay the filter segment, sample surface upward, on top of the solution. Bring the filter and solution together at an angle of about 20° to help exclude air bubbles. Remove any excess clearing solution. Place the slide in an oven or on a hot plate, in a fume hood, at 65 to 70°C for 10 min.

12.3 Plasma etching of the collapsed filter is required.

12.3.1 The microscope slide to which the collapsed filter pieces are attached is placed in a plasma asher (7.27). Because plasma ashers vary greatly in their performance, both from unit to unit and between different positions in the asher chamber, it is difficult to specify the exact conditions that must be used. Insufficient etching will result in a failure to expose embedded fibers, and too much etching may result in the loss of particles from the filter surface. To determine the optimum time for ashing, place an unused 25 mm diameter MCE filter in the center of a glass microscope slide. Position the slide approximately in the center of the asher chamber. Close the chamber and evacuate to a pressure of approximately 40 Pa, while admitting oxygen to the chamber at a rate of 8 to 20 cm^3/min. Adjust the tuning of the system so that the intensity of the plasma is maximized. Determine the time required for complete oxidation of the filter. Adjust the system parameters to achieve complete oxidation of the filter in a period of approximately 15 min. For etching of collapsed filters, use these operating parameters for a period of 8 min. For additional information on calibration, see the *USEPA Asbestos-Containing Materials in Schools* (4) or *NIST/NVLAP Program Handbook for Airborne Asbestos Analysis* (6) documents.

12.3.2 Place the glass slide containing the collapsed filters into the low-temperature plasma asher, and etch the filter.

12.4 Carbon coating of the collapsed and etched filters is required.

12.4.1 Carbon coating must be performed with a high-vacuum coating unit (7.4), capable of less than 10^{-4} torr (13 MPa) pressure. Units that are based on evaporation of carbon filaments in a vacuum generated only by an oil rotary pump have not been evaluated for this application and shall not be used. Carbon rods (7.40) used for evaporators shall be sharpened with a carbon rod sharpener to a neck of about 4 mm in length and 1 mm in diameter. The rods are installed in the evaporator in such a manner that the points are approximately 100 to 120 mm from the surface of the microscope slide held in the rotating device.

12.4.2 Place the glass slide holding the filters on the rotation device, and evacuate the evaporator chamber to a vacuum of at least 13 MPa. Perform the evaporation in very short bursts, separated by 3 to 4 s to allow the electrodes to cool. An alternate method of evaporation is by using a slow continuous applied current. An experienced analyst can judge the thickness of the carbon film to be applied. Conduct tests on unused filters first. If the carbon film is too thin, large particles will be lost from the TEM specimen, and there will be few complete and undamaged grid openings on the specimen.

12.4.2.1 If the coating is too thick, it will lead to a TEM image that is lacking in contrast, and the ability to obtain electron diffraction patterns will be compromised. The carbon film shall be as thin as possible and still remain intact on most of the grid openings of the TEM specimen.

12.5 *Preparation of the Jaffe Washer*— The precise design of the Jaffe washer is not considered important, so any one of the published designs may be used (7, 8). One such washer consists of a simple stainless steel bridge contained in a glass petri dish.

12.5.1 Place several pieces of lens tissue (7.41) on the stainless steel bridge. The pieces of lens tissue shall be large enough to completely drape over the bridge and into the solvent. In a fume hood, fill the petri dish with acetone (or DMF) until the height of the solvent is brought up to contact the underside of the metal bridge as illustrated in Fig. 2.

12.6 *Placing the Specimens into the Jaffe Washer*:

12.6.1 Place the TEM grids (7.39) shiny side up on a piece of lens tissue or filter paper so that individual grids can be easily picked up with tweezers.

12.6.2 Prepare three grids from each sample.

12.6.2.1 Using a curved scalpel blade (7.20), excise at least two square (3 mm by 3 mm) pieces of the carbon-coated MCE filter from the glass slide.

12.6.2.2 Place the square filter piece carbon-side up on top of a TEM specimen grid.

12.6.2.3 Place the whole assembly (filter/grid) on the saturated lens tissue in the Jaffe washer.

12.6.2.4 Place the three TEM grid sample filter preparations on the same piece of lens tissue in the Jaffe washer.

12.6.2.5 Place the lid on the Jaffe washer and allow the system to stand for several hours.

12.7 Alternately, place the grids on a low level (petri dish filled to the ⅛ mark) DMF Jaffe washer for 60 min. Add enough solution of equal parts DMF/acetone to fill the washer to the screen level. Remove the grids after 30 min if they have cleared, that is, all filter material has been removed from the carbon film, as determined by inspection in the TEM.

12.8 Carefully remove the grids from the Jaffe washer, allowing the grids to dry before placing them in a clean marked grid box.

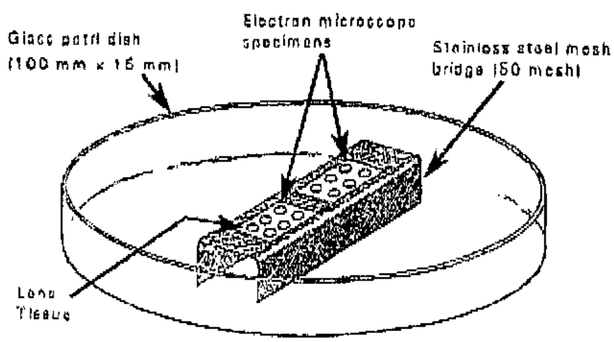


FIG. 2 Example of Design of Solvent Washer (Jaffe Washer)

13. TEM Specimen Preparation of Polycarbonate (PC) Filter

13.1 Cover the surface of a clean microscope slide with two strips of double-sided adhesive tape.

13.2 Cut a strip of filter paper slightly narrower than the width of the slide. Position the filter paper strip on the center of the length of the slide.

13.3 Using a clean, curved scalpel blade, cut a strip of the PC filter approximately 25 by 6 mm. Use a rocking motion of the scalpel blade to avoid tearing the filter. Place the PC strip particle side up on the slide perpendicular to the long axis of the slide. The ends of the PC strip must contact the double sided adhesive tape. Each slide can hold several PC strips. With a glass marker, label each PC strip with the individual sample number.

13.4 Carbon coat the PC filter strips as discussed in 12.4.2. PC filters do not require etching.

NOTE 2—**Caution**: Do not overheat the filter sections while carbon coating.

13.5 Prepare a Jaffe washer as described in 12.5, but fill the washer with chloroform or 1-methyl-2-pyrrolidone to the level of the screen.

13.6 Using a clean curved scalpel blade, excise three, 3-mm square filter pieces from each PC strip. Place the filter squares carbon side up on the shiny side of a TEM grid. Pick up the grid and filter section together and place them on the lens tissue in the Jaffe washer.

13.7 Place the lid on the Jaffe washer and rest the grids in place for at least 4 h. Best results are obtained with longer wicking times, up to 12 h.

13.8 Carefully remove the grids from the Jaffe washer, allowing the grids to dry before placing them in a clean, marked grid box.

14. Grid Opening Measurements

14.1 TEM grids must have a known grid opening area. Determine this area as follows:

14.2 Measure at least 20 grid openings in each of 20 random 75 to 100 μm (200-mesh) copper grids for a total of 400 grid openings for every 1000 grids used, by placing the 20 grids on a glass slide and examining them under the optical microscope. Use a calibrated graticule to measure the average length and width of the 20 openings from each of the individual grids. From the accumulated data, calculate the average grid opening area of the 400 openings.

14.3 Grid area measurements can also be made at the TEM at a calibrated screen magnification of between 15 000 and 20 000X. Typically measure one grid opening for each grid examined. Measure grid openings in both the x and y directions and calculate the area.

14.4 Pre-calibrated TEM grids are also acceptable for this test method.

15. TEM Method

15.1 Microscope settings: 80 to 120 kV, 15 000 to 20 000X screen magnification for analysis (7.2).

15.2 Analyze two grids for each sample. Analyze one-half of the sample area on one sample grid preparation and the remaining half on a second sample grid preparation.

15.3 *Determination of Specimen Suitability:*

15.3.1 Carefully load the TEM grid, carbon side facing up (in the TEM column) with the grid bars oriented parallel/perpendicular to the length of the specimen holder. Use a hand lens or loupe, if necessary. This procedure will line up the grid with the *x* and *y* translation directions of the microscope. Insert the specimen holder into the microscope.

15.3.2 Scan the entire grid at low magnification (250X to 1000X) to determine its suitability for high magnification analysis as specified in 15.3.3.

15.3.3 Grids are acceptable for analysis if the following conditions are met:

15.3.3.1 The fraction of grid openings covered by the replica section is at least 50 %.

15.3.3.2 Relative to that section of the grid covered by the carbon replica, the fraction of intact grid openings is greater than 50 %.

15.3.3.3 The fractional area of undissolved filter is less than 10 %.

15.3.3.4 The fraction of grid openings with overlapping or folded replica film is less than 50 %.

15.3.3.5 At least 20 grid openings, that have no overlapping or folded replica, are less than 5 % covered with holes and have less than 5 % opaque area due to incomplete filter dissolution.

15.4 *Determination of Grid Opening Suitability:*

15.4.1 If the grid meets acceptance criteria, choose a grid opening for analysis from various areas of the grid so that the entire grid is represented. Determine the suitability of each individual grid opening prior to the analysis.

15.4.2 The individual grid opening must have less than 5 % holes over its area.

15.4.3 Grid openings must be less than 25 % covered with particulate matter.

15.4.4 Grid openings must be uniformly loaded.

15.5 Observe and record the orientation of the grid at 80 to 150X, on a grid map record sheet along with the location of the grid openings that are examined for the analysis. If indexed grids are used, a grid map is not required, but the identifying coordinates of the grid square must be recorded.

16. Recording Data Rules

16.1 Record on the count sheet any continuous grouping of particles in which an asbestos fiber is detected. Classify asbestos structures as fibers, bundles, clusters, or matrices as defined in 5.2.

16.2 Use the criteria for fiber, bundle, cluster, and matrix identification, as described in the *USEPA Asbestos-Containing Materials in Schools* document (4). Record, for each AHERA structure identified, the length and width measurements.

16.3 Record NSD (No Structures Detected) when no structures are detected in the grid opening.

16.4 Identify structures classified as chrysotile identified by either electron diffraction or X-ray analysis (7.3) and recorded on a count sheet. Verify at least one out of every ten chrysotile structures by X-ray analysis.

16.5 Structures classified as amphiboles by X-ray analysis and electron diffraction are recorded on the count sheet. For more information on identification, see Yamate, et al, (7) or Chatfield and Dillon (8).

16.6 Record a typical electron diffraction pattern for each type of asbestos observed for each group of samples (or a minimum of every five samples) analyzed. Record the micrograph number on the count sheet. Record at least one X-ray spectrum for each type of asbestos observed per sample. Attach the print-outs to the back of the count sheet. If the X-ray spectrum is stored, record the file and disk number on the count sheet.

16.7 *Counting Rules:*

16.7.1 At a screen magnification of between 15 000 and 20 000X evaluate the grids for the most concentrated sample loading; reject the sample if it is estimated to contain more than 50 asbestos structures per grid opening. Proceed to the next lower concentrated sample until a set of grids are obtained that have less than 30 asbestos structures per grid opening.

16.8 *Analytical Sensitivity (AS)*—As determined by the following equation:

$$(EFA \times 100\ mL \times 1)/(GO \times GOA \times V \times SPL) = AS \qquad (1)$$

where:

EFA = effective filter area of the final sampling filter, mm^2
GO = number of grid openings counted
GOA = average grid opening area, mm^2
SPL = surface area sampled, cm^2
V = volume of sample filtered in step 10.4.9, representing the actual volume taken from the original 100 mL suspension, mL
AS = analytical sensitivity, expressed as asbestos structures/cm^2

16.8.1 An AS of approximately 1000 asbestos structures per square centimetre (calculated for the detection of a single asbestos structure) has been designed for this analysis. This sensitivity can be achieved by increasing the amount of liquid filtered, increasing the number of grid openings analyzed, increasing the area of the collection, or decreasing the size of the final filter. For example, using a collection area = 500 cm^2, filter area = 1000 mm^2, number of grid openings = 10, and a grid area of 0.01 mm^2, V = 50 mL, the AS is 40 str/cm^2. Occasionally, due to high particle loadings or high asbestos surface loading, this AS cannot be practically achieved and stopping rules apply.

16.8.2 The numerical value of AS required for any specific application of this method may be achieved by selecting an appropriate combination of the sampling and analysis parameters in the above equation. For example, if SPL = 100 cm^2, EFA = 1000 mm^2, GO = 10, GOA = 0.01 mm^2, V = 10 mL, and D = 1, then AS = 1000 str/cm^2. Increasing GO to 50 and V to 50 mL changes the AS to 40 Str/cm^2.

16.8.3 When sample filters are heavily loaded with particulate matter, it may useful to employ serial dilutions during preparation to reduce the loading on grid specimens to acceptable levels and thus facilitate analysis. Under such circumstances, the AS may be calculated by substituting an appropriate value for the dilution factor, D, into the above equation. In general:

$$D = VA/(V + VPFW) \quad (2)$$

VA = the volume of the aliquot from the new, diluted suspension that is filtered to prepare the analytical filter; V = the volume of the aliquot from the initial (100 mL) suspension that is diluted; and VPFW = the volume of particle free water added to V during serial dilution to produce the new, diluted suspension.

Thus, if GO = 10, V = 10 mL, VPFW = 90 mL, and VA = 1.0 mL, D = 0.01 and the AS = 100 000 str/cm^2.

16.9 *Limit of Detection*—The limit of detection for this test method is calculated using the Practice D 6620. All data shall be provided in the laboratory report.

16.10 *Stopping Rules*:

16.10.1 The analysis is stopped upon the completion of the grid square that achieves an AS of less than 1000 asbestos structures per square centimetre.

16.10.2 If an AS of 1000 asbestos structures per square centimetre cannot be achieved after analyzing ten grid openings then stop on grid opening No. 10 or the grid opening which contains the 100th asbestos structure, whichever comes first. A minimum of four grid squares shall be analyzed for each sample.

16.10.2.1 If the analysis is stopped because of the 100th structure rule, the entire grid square containing the 100th structure must be counted.

16.11 After analysis, remove the grids from the TEM, and replace them in the appropriate grid storage holder.

17. Sample Storage

17.1 The washed-out sample cassettes can be discarded after use.

17.2 Sample grids and unused filter sections (7.18) must be stored for a minimum of one year.

18. Reporting

18.1 Report the following information for each dust sample analyzed:

18.1.1 Surface loading in structures/cm^2.

18.1.2 The AS.

18.1.3 Types of asbestos present.

18.1.4 Number of asbestos structures counted.

18.1.5 Effective filtration area.

18.1.6 Average size of the TEM grid openings that were counted.

18.1.7 Number of grid openings examined.

18.1.8 Sample dilution used.

18.1.9 Area of the surface sampled.

18.1.10 Listing of size data for each structure counted.

18.1.11 A copy of the TEM count sheet or a complete listing of the raw data. An example of a typical count sheet is shown in Appendix X1.

18.2 Determine the amount of asbestos in any accepted sample using the following formula:

$$\frac{EFA \times 100\ \text{mL} \times \#STR}{GO \times GOA \times V \times SPL} = \text{asbestos structures/cm}^2 \quad (3)$$

where:

$\#STR$ = number of asbestos structures counted,
EFA = effective filter area of the final sampling filter, mm^2,
GO = number of grid openings counted,
GOA = average grid opening area, mm^2,
SPL = surface area sampled, cm^2, and
V = volume of sample filtered in step 10.4.9, representing the actual volume taken from the original 100 mL suspension, mL.

19. Quality Control/Quality Assurance

19.1 In general, the laboratory's quality control checks are used to verify that a system is performing according to specifications regarding accuracy and consistency. In an analytical laboratory, spiked or known quantitative samples are normally used. However, due to the difficulties in preparing known quantitative asbestos samples, routine quality control testing focuses on re-analysis of samples (duplicate recounts).

19.1.1 Re-analyze samples at a rate of 1/10 of the sample sets (one out of every ten samples analyzed not including laboratory blanks). The re-analysis shall consist of a second sample preparation obtained from the final filter.

19.2 In addition, quality assurance programs must follow the criteria shown in the *USEPA Asbestos-Containing Materials in Schools* document (4) and in the *NIST/NVLAP Program Handbook for Airborne Asbestos Analysis* document (6). These documents describe sample custody, sample preparation, blank checks for contamination, calibration, sample analysis, analyst qualifications, and technical facilities.

20. Calibrations

20.1 Perform calibrations of the instrumentation on a regular basis, and retain these records in the laboratory, in accordance with the laboratory's quality assurance program.

20.2 Record calibrations in a log book along with dates of calibration and the attached backup documentation.

20.3 A calibration list for the instrument is as follows:

20.3.1 *TEM*:

20.3.1.1 Check the alignment and the systems operation. Refer to the TEM manufacturer's operational manual for detailed instructions.

20.3.1.2 Calibrate the camera length of the TEM in electron diffraction (ED) operating mode before ED patterns of unknown samples are observed. Camera length can be measured by using a carbon coated grid on which a thin film of gold has been sputtered or evaporated. A thin film of gold is evaporated on the specimen TEM grid to obtain zone-axis ED patterns superimposed with a ring pattern from the polycrystalline gold film. In practice, it is desirable to optimize the thickness of the gold film so that only one or two sharp rings are obtained on the superimposed ED pattern. Thick gold films will tend to mask weak diffraction spots from the fibrous particles. Since the unknown d-spacings of most interest in asbestos analysis are those which lie closest to the transmitted beam, multiple gold rings from thick films are unnecessary. Alternatively, a gold standard specimen can be used to obtain an average camera constant calculated for that particular instrument and can then be used for ED patterns of unknowns taken during the corresponding period.

20.3.1.3 Perform magnification calibration at the fluorescent screen. This calibration must be performed at the magnification used for structure counting. Calibration is performed with a grating replica (7.47) (for example, one containing at least 2160 lines/mm).

(a) Define a field of view on the fluorescent screen. The field of view must be measurable or previously inscribed with a scale or concentric circles (all scales should be metric).

(b) Frequency of calibration will depend on the service history of the particular microscope.

(c) Check the calibration after any maintenance of the microscope that involves adjustment of the power supply to the lens or the high voltage system or the mechanical disassembly of the electron optical column (apart from filament exchange).

(d) The analyst must ensure that the grating replica is placed at the same distance from the objective lens as the specimen.

(e) For instruments that incorporate a eucentric tilting specimen stage, all specimens and the grating replica must be placed at the eucentric position.

20.3.1.4 The smallest spot size of the TEM must be checked.

(a) At the crossover point, photograph the spot size at a screen magnification of 15 000 to 20 000X. An exposure time of 1 s is usually adequate.

(b) The measured spot size must be less than or equal to 250 nm.

20.4 *EDXA*:

20.4.1 The resolution and calibration of the EDXA must be verified.

20.4.1.1 Collect a standard EDXA Cu peak from the Cu grid.

20.4.1.2 Compare the X-ray energy versus channel number for the Cu peak and be certain that readings are within ±10 eV.

20.4.2 Collect a standard EDXA of crocidolite asbestos (NIST SRM 1866).

20.4.2.1 The elemental analysis of the crocidolite must resolve the Na peak.

20.4.3 Collect a standard EDXA of chrysotile asbestos.

20.4.3.1 The elemental analysis of chrysotile must resolve both Si and Mg on a single chrysotile fiber.

20.5 Ultrasonic bath calibration shall be performed as follows:

20.5.1 Fill the bath water to a level equal to the height of suspension in the glass sample container that will be used for the dust analysis. Operate the bath until the water reaches the equilibrium temperature.

20.5.2 Place 100 mL of water (at approximately 20°C) in another 200-mL glass sample container, and record its temperature.

20.5.3 Place the sample container in the water in the ultrasonic bath (with the power turned off). After 60 s, remove the glass container and record its temperature.

20.5.4 Place 100 mL of water (at approximately 20°C) in another 200-mL glass sample container, and record its temperature.

20.5.5 Place the second sample container into the water in the ultrasonic bath (with the power turned on). After 60 s, remove the glass container and record its temperature.

20.5.6 Calculate the rate of energy deposition into the sample container using the following formula:

$$R = 4.185 \times \sigma \times \rho \times \frac{(\theta_2 - \theta_1)}{t} \tag{4}$$

where:

4.185	=	Joules/cal,
R	=	energy deposition, watts/mL,
θ_1	=	temperature rise with the ultrasonic bath not operating, °C,
θ_2	=	temperature rise with the ultrasonic bath operating, °C,
t	=	time in seconds, 60 s (20.5.3 and 20.5.5),
σ	=	specific heat of the liquid in the glass sample container, 1.0 cal/g, and
ρ	=	density of the liquid in the glass sample container, 1.0 g/cm^3.

20.5.7 Adjust the operating conditions of the bath so that the rate of energy deposition is in the range of 0.08 to 0.12 MW/m^3, as defined by this procedure.

21. Precision and Bias

21.1 *Precision*—The precision of the procedure in this test method is being determined using round robin data from participating laboratories.

21.2 *Bias*—Since there is no accepted reference material suitable for determining the bias of the procedure in this test method, bias has not been determined (see Specification D 3670).

NOTE 3—Round robin data is under development and will be presented as a research report.

22. Keywords

22.1 asbestos; microvacuuming; settled dust; TEM

APPENDIX

(Nonmandatory Information)

X1. DUST SAMPLE ANALYSIS

X1.1 See Figs. X1.1 and X1.2 for the dust analysis worksheet and the TEM count sheet.

DUST SAMPLE ANALYSIS

Client:	____________	Accelerating Voltage:	____________
Sample ID:	____________	Indicated Mag:	KX
Job Number:	____________	Screen Mag:	KX
Date Sample Analyzed:	- -	Microscope:	1 2 3 4 5
Number of Openings/Grids Counted:	|	Filter Type:	____________
Grid Accepted, 600X:	Yes No	Filter Size:	____________
Percent Loading:	%	Filter Pore Size (μm):	____________
Grid Box #1:	____________	Grid Opening:	1) μm x μm
			2) μm x μm

Analyst: ____________

Reviewer: ____________ Counting Rules: AHERA LEVEL II

Calculation Data:

Effective Filter Area in mm²:	(EFA)	____________
Number of Grid Openings Counted:	(GO)	____________
Average Grid Opening Area in mm²:	(GOA)	____________
Volume of sample Filtered in ml:	(V)	____________
Surface area Sampled in cm²:	(SPL)	____________
Number of Asbestos Structures Counted:*	(#STR)	____________

* If the number of asbestos structures counted is less than or equal to 4, enter 4 structures as the limit of detection here.

FORMULA FOR CALCULATION OF ASBESTOS STRUCTURES "DUST" PER CM²:

$$\frac{EFAX \ X \ 100 \ X \ \#STR}{GO \ X \ GOA \ X \ VX \ SPL} = \text{(Asbestos Structures per cm}^2\text{)}$$

Results for Total Asbestos Structures: ____________
(Structures per cm²)

Results for Structures ≥ microns: ____________
(Structures per cm²)

FIG. X1.1 Dust Sample Analysis Work Sheet

Job Number: ______________________

Structure #	Grid # Square #	Type	Structure	Length Microns	Width Microns	Confirmation		
						Morph.	SAED	EDS

Note: Keys to Abbreviations Used in Figure:

Type:

C = Chrysotile
AM = Amosite
CR = Crocidolite
AC = Actinolite
TR = Tremolite
AN = Anthophyllite
N = Non Asbestos

Structure:

F = Fiber
B = Bundle
C = Cluster
M = Matrix

Others:

NSD = No Structures Detected
Morph = Morphology
SAED = Selected Area Electron Diffraction
EDS = Energy Dispersive X-Ray Spectroscopy
ER = Inter-Row Spacing
NP = No Pattern

FIG. X1.2 TEM Count Sheet

REFERENCES

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(3) Leake, B. E., "Nomenclature of Amphiboles," *American Mineralogist*, Vol 63, 1978, pp. 1023–1052.

(4) " *USEPA Asbestos-Containing Materials in Schools: Final Rule and Notice,*" *Federal Register*, 40 CFR Part 763, Appendix A to Sub-part E., Oct. 30, 1987.

(5) OSHA, *OSHA Technical Manual, OSHA Instruction CPL 2-20B*, Directorate of Technical Support, U.S. Department of Labor, Washington, DC 20210, Feb. 5, 1990, pp. 1–8 to 1–11.

(6) *NIST/NVLAP Program Handbook for Airborne Asbestos Analysis*, NISTIR, August 1989, pp. 89–4137.

(7) Yamate, G., Agarwall, S. C., and Gibbons, R. D., "Methodology for the Measurement of Airborne Asbestos by Electron Microscopy," EPA Draft Report, Contract No. 68-02-3266, 1984.

(8) Chatfield, E. J., and Dillon, M. J., "Analytical Method for the Determination of Asbestos in Water," EPA No. 600/4-83-043, 1983.

APPENDIX H:
ISO Method 10312:1995

Designation: D 5755 – 03

Standard Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Structure Number Surface Loading[1]

This standard is issued under the fixed designation D 5755; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers a procedure to (*a*) identify asbestos in dust and (*b*) provide an estimate of the surface loading of asbestos in the sampled dust reported as the number of asbestos structures per unit area of sampled surface.

1.1.1 If an estimate of the asbestos mass is to be determined, the user is referred to Test Method D 5756.

1.2 This test method describes the equipment and procedures necessary for sampling, by a microvacuum technique, non-airborne dust for levels of asbestos structures. The non-airborne sample is collected inside a standard filter membrane cassette from the sampling of a surface area for dust which may contain asbestos.

1.2.1 This procedure uses a microvacuuming sampling technique. The collection efficiency of this technique is unknown and will vary among substrates. Properties influencing collection efficiency include surface texture, adhesiveness, electrostatic properties and other factors.

1.3 Asbestos identified by transmission electron microscopy (TEM) is based on morphology, selected area electron diffraction (SAED), and energy dispersive X-ray analysis (EDXA). Some information about structure size is also determined.

1.4 This test method is generally applicable for an estimate of the surface loading of asbestos structures starting from approximately 1000 asbestos structures per square centimetre.

1.4.1 The procedure outlined in this test method employs an indirect sample preparation technique. It is intended to disperse aggregated asbestos into fundamental fibrils, fiber bundles, clusters, or matrices that can be more accurately quantified by transmission electron microscopy. However, as with all indirect sample preparation techniques, the asbestos observed for quantification may not represent the physical form of the asbestos as sampled. More specifically, the procedure described neither creates nor destroys asbestos, but it may alter the physical form of the mineral fibers.

1.5 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water[2]

D 3195 Practice for Rotameter Calibration[3]

D 3670 Guide for Determination of Precision and Bias of Methods of Committee D22[3]

D 5756 Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Mass Surface Loading[3]

D 6620 Practice for Determining a Detection Limit for Asbestos Measurements Based on Counts[3]

3. Terminology

3.1 *Definitions:*

3.1.1 *asbestiform*—a special type of fibrous habit in which the fibers are separable into thinner fibers and ultimately into fibrils. This habit accounts for greater flexibility and higher tensile strength than other habits of the same mineral. For more information on asbestiform mineralogy, see Refs **(1)**,[4] **(2)** and **(3)**.

3.1.2 *asbestos*—a collective term that describes a group of naturally occurring, inorganic, highly fibrous, silicate dominated minerals, which are easily separated into long, thin, flexible fibers when crushed or processed.

[1] This test method is under the jurisdiction of ASTM Committee D22 on Sampling and Analysis of Atmospheres and is the direct responsibility of Subcommittee D22.07 on Sampling and Analysis of Asbestos.

Current edition approved April 10, 2003. Published June 2003. Originally approved in 1995. Last previous edition approved in 2002 as D 5755 - 02.

[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] *Annual Book of ASTM Standards*, Vol 11.03.

[4] The boldface numbers in parentheses refer to the list of references at the end of this test method.

3.1.2.1 *Discussion*—Included in the definition are the asbestiform varieties of: serpentine (chrysotile); riebeckite (crocidolite); grunerite (grunerite asbestos); anthophyllite (anthophyllite asbestos); tremolite (tremolite asbestos); and actinolite (actinolite asbestos). The amphibole mineral compositions are defined according to nomenclature of the International Mineralogical Association **(3)**.

Asbestos	Chemical Abstract Service No.[5]
Chrysotile	12001-29-5
Crocidolite	12001-28-4
Grunerite Asbestos	12172-73-5
Anthophyllite Asbestos	77536-67-5
Tremolite Asbestos	77536-68-6
Actinolite Asbestos	77536-66-4

3.1.3 *fibril*—a single fiber that cannot be separated into smaller components without losing its fibrous properties or appearance.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *aspect ratio*—the ratio of the length of a fibrous particle to its average width.

3.2.2 *bundle*—a structure composed of three or more fibers in a parallel arrangement with the fibers closer than one fiber diameter to each other.

3.2.3 *cluster*—a structure with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated from the group; groupings of fibers must have more than two points touching.

3.2.4 *debris*—materials that are of an amount and size (particles greater than 1 mm in diameter) that can be visually identified as to their source.

3.2.5 *dust*—any material composed of particles in a size range of <1 mm.

3.2.6 *fiber*—a structure having a minimum length of 0.5 μm, an aspect ratio of 5:1 or greater, and substantially parallel sides **(4)**.

3.2.7 *fibrous*—of a mineral composed of parallel, radiating, or interlaced aggregates of fibers, from which the fibers are sometimes separable. That is, the crystalline aggregate may be referred to as fibrous even if it is not composed of separable fibers, but has that distinct appearance. The term fibrous is used in a general mineralogical way to describe aggregates of grains that crystallize in a needle-like habit and appear to be composed of fibers. Fibrous has a much more general meaning than asbestos. While it is correct that all asbestos minerals are fibrous, not all minerals having fibrous habits are asbestos.

3.2.8 *indirect preparation*—a method in which a sample passes through one or more intermediate steps prior to final filtration.

3.2.9 *matrix*—a structure in which one or more fibers, or fiber bundles that are touching, are attached to, or partially concealed by a single particle or connected group of nonfibrous particles. The exposed fiber must meet the fiber definition (see 3.2.6).

3.2.10 *structures*—a term that is used to categorize all the types of asbestos particles which are recorded during the analysis (such as fibers, bundles, clusters, and matrices). Final results of the test are always expressed in asbestos structures per square centimetre.

4. Summary of Test Method

4.1 The sample is collected by vacuuming a known surface area with a standard 25 or 37 mm air sampling cassette using a plastic tube that is attached to the inlet orifice which acts as a nozzle. The sample is transferred from inside the cassette to an aqueous suspension of known volume. Aliquots of the suspension are then filtered through a membrane. A section of the membrane is prepared and transferred to a TEM grid using the direct transfer method. The asbestiform structures are identified, sized, and counted by TEM, using SAED and EDXA at a magnification of 15 000 to 20 000X.

5. Significance and Use

5.1 This microvacuum sampling and indirect analysis method is used for the general testing of non-airborne dust samples for asbestos. It is used to assist in the evaluation of dust that may be found on surfaces in buildings such as ceiling tiles, shelving, electrical components, duct work, carpet, etc. This test method provides an index of the surface loading of asbestos structures in the dust per unit area analyzed as derived from a quantitative TEM analysis.

5.1.1 This test method does not describe procedures or techniques required to evaluate the safety or habitability of buildings with asbestos-containing materials, or compliance with federal, state, or local regulations or statutes. It is the user's responsibility to make these determinations.

5.1.2 At present, no relationship has been established between asbestos-containing dust as measured by this test method and potential human exposure to airborne asbestos. Accordingly, the users should consider other available information in their interpretation of the data obtained from this test method.

5.2 This definition of dust accepts all particles small enough to pass through a 1 mm (No. 18) screen. Thus, a single, large asbestos containing particle(s) (from the large end of the particle size distribution) dispersed during sample preparation may result in anomalously large asbestos surface loading results in the TEM analyses of that sample. It is, therefore, recommended that multiple independent samples are secured from the same area, and that a minimum of three samples be analyzed by the entire procedure.

6. Interferences

6.1 The following minerals have properties (that is, chemical or crystalline structure) which are very similar to asbestos minerals and may interfere with the analysis by causing a false positive to be recorded during the test. Therefore, literature references for these materials must be maintained in the laboratory for comparison to asbestos minerals so that they are not misidentified as asbestos minerals.

6.1.1 *Antigorite.*

6.1.2 *Palygorskite (Attapulgite).*

6.1.3 *Halloysite.*

6.1.4 *Pyroxenes.*

6.1.5 *Sepiolite.*

6.1.6 *Vermiculite scrolls.*

[5] The non-asbestiform variations of the minerals indicated in 3.1.3 have different Chemical Abstract Service (CAS) numbers.

6.1.7 *Fibrous talc.*

6.1.8 Hornblende and other amphiboles other than those listed in 3.1.2.

6.2 Collecting any dust particles greater than 1 mm in size in this test method may cause an interference and, therefore, must be avoided.

7. Materials and Equipment

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.[6]

7.2 *Transmission Electron Microscope (TEM)*, an 80 to 120 kV TEM, capable of performing electron diffraction, with a fluorescent screen inscribed with calibrated gradations, is required. The TEM must be equipped with energy dispersive X-ray spectroscopy (EDXA) and it must have a scanning transmission electron microscopy (STEM) attachment or be capable of producing a spot size of less than 250 nm in diameter in crossover.

7.3 *Energy Dispersive X-ray System (EDXA).*

7.4 *High Vacuum Carbon Evaporator*, with rotating stage.

7.5 *High Efficiency Particulate Air (HEPA)*, filtered negative flow hood.

7.6 *Exhaust or Fume Hood.*

7.7 *Particle-free Water* (ASTM Type II, see Specification D 1193).

7.8 *Glass Beakers* (50 mL).

7.9 *Glass Sample Containers*, with wide mouth screw cap (200 mL) or equivalent sealable container (height of the glass sample container should be approximately 13 cm high by 6 cm wide).

7.10 *Waterproof Markers.*

7.11 *Forceps* (tweezers).

7.12 *Ultrasonic Bath*, table top model (100 W).

7.13 *Graduated Pipettes* (1, 5, 10 mL sizes), glass or plastic.

7.14 *Filter Funnel*, either 25 mm or 47 mm, glass or disposable. Filter funnel assemblies, either glass or disposable plastic, and using either a 25 mm or 47 mm diameter filter.

7.15 *Side Arm Filter Flask*, 1000 mL.

7.16 *Mixed Cellulose Ester (MCE) Membrane Filters*, 25 or 47 mm diameter, $\leq$0.22 µm and 5 µm pore size.

7.17 *Polycarbonate (PC) Filters*, 25 or 47 mm diameter, $\leq$0.2 µm pore size.

7.18 *Storage Containers*, for the 25 or 47 mm filters (for archiving).

7.19 *Glass Slides*, approximately 76 by 25 mm in size.

7.20 *Scalpel Blades*, No. 10, or equivalent.

7.21 *Cabinet-type Desiccator*, or low temperature drying oven.

7.22 *Chloroform*, reagent grade.

7.23 *Acetone*, reagent grade.

7.24 *Dimethylformamide (DMF).*

7.25 *Glacial Acetic Acid.*

7.26 *1-methyl-2-pyrrolidone.*

7.27 *Plasma Asher*, low temperature.

7.28 *pH Paper.*

7.29 *Air Sampling Pump*, low volume personal-type, capable of achieving a flow rate of 1 to 5 L/min.

7.30 *Rotameter.*

7.31 *Air Sampling Cassettes*, 25 mm or 37 mm, containing 0.8µ m or smaller pore size MCE or PC filters.

7.32 *Cork Borer*, 7 mm.

7.33 *Non-Asbestos Mineral*, references as outlined in 6.1.

7.34 *Asbestos Standards*, as outlined in 3.1.2.

7.35 *Tygon*[7] *Tubing*, or equivalent.

7.36 *Small Vacuum Pump*, that can maintain a pressure of 92 kPa.

7.37 *Petri Dishes*, large glass, approximately 90 mm in diameter.

7.38 *Jaffe Washer*, stainless steel or aluminum mesh screen, 30 to 40 mesh, and approximately 75 mm by 50 mm in size.

7.39 *Copper TEM Finder Grids*, 200 mesh.

7.40 *Carbon Evaporator Rods.*

7.41 *Lens Tissue.*

7.42 *Ashless Filter Paper Filters*, 90 mm diameter.

7.43 *Gummed Paper Reinforcement Rings.*

7.44 *Wash Bottles*, plastic.

7.45 *Reagent Alcohol*, HPLC Grade (Fisher A995 or equivalent).

7.46 *Opening Mesh Screen*, plastic, 1.0 by 1.0 mm, (Spectra-Mesh #146410 or equivalent).

7.47 *Diffraction Grating Replica.*

8. Sampling Procedure for Microvacuum Technique

8.1 For sampling asbestos-containing dust in either indoor or outdoor environments, commercially available cassettes must be used. Air monitoring cassettes containing 25 mm or 37 mm diameter mixed cellulose ester (MCE) or polycarbonate (PC) filter membranes with a pore size less than or equal to 0.8 µm are required (7.31). The number of samples collected depends upon the specific circumstances of the study.

8.2 Maintain a log of all pertinent sampling information and sampling locations.

8.3 Sampling pumps and flow indicators shall be calibrated using a certified standard apparatus or assembly (see Practice D 3195 and 7.29).

8.4 Record all calibration information **(5)**.

8.5 Perform a leak check of the sampling system at each sampling site by activating the pump (7.29) with the closed sampling cassette in line. Any air flow shows that a leak is present that must be eliminated before initiating the sampling operation.

[6] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

[7] Tygon is a registered trademark of the DuPont Co.

8.6 Attach the sampling cassette to the sampling pump at the outlet side of the cassette with plastic tubing (7.35). The plastic tubing must be long enough in that the sample areas can be reached without interference from the sampling pump. Attach a clean, approximately 25.4 mm long piece of plastic tubing (6.35 mm internal diameter) directly to the inlet orifice. Use this piece of tubing as the sampling nozzle. Cut the sampling end of the tubing at a 45° angle as illustrated in Fig. 1. The exact design of the nozzle is not critical as long as some vacuum break is provided to avoid simply pushing the dust around on the surface with the nozzle rather than vacuuming it into the cassette. The internal diameter of the nozzle and flow rate of the pump may vary as long as the air velocity is 100 (±10) cm/s. This air velocity calculation is based on an internal sampling tube diameter of 6.35 mm at a flow rate of 2 L/min.

8.7 Measure and determine the sample area of interest. A sample area of 100 cm^2 is vacuumed until there is no visible dust or particulates matter remaining. Perform a minimum of two orthogonal passes on the surface within a minimum of 2 min of sampling time. Avoid scraping or abrading the surface being sampled. (Do not sample any debris or dust particles greater than 1 mm in diameter (see 4.2).) Smaller or larger areas can be sampled, if needed. For example, some surfaces of interest may have a smaller area than 100 cm^2. Less dusty surfaces may require vacuuming of larger areas. Unlike air samples, the overloading of the cassettes with dust will not be a problem. As defined in 3.2.5, only dust shall be collected for this analysis.

8.8 At the end of sample collection, invert the cassette so that the nozzle inlet faces up before shutting off the power to the pump. The nozzle is then sealed with a cassette end-plug and the cassette/nozzle taped or appropriately packaged to prevent separation of the nozzle and cassette assembly. A second option is the removal of the nozzle from the cassette, then plugging of the cassette and shipment of the nozzle (also plugged at both ends) sealed in a separate closeable plastic bag. A third option is placing the nozzle inside the cassette for shipment. The nozzle is always saved and rinsed because a significant percentage of the dust drawn from a lightly loaded surface may adhere to the inside walls of the tubing.

8.9 Check that all samples are clearly labeled, that all dust sampling information sheets are completed, and that all pertinent information has been enclosed, in accordance with laboratory quality control practices, before transfer of the samples to the laboratory. Include an unused cassette and nozzle as a field blank.

8.10 Wipe off the exterior surface of the cassettes with disposable wet towels (baby wipes) prior to packaging for shipment.

9. Sample Shipment

9.1 Ship dust samples to an analytical laboratory in a sealed container, but separate from any bulk or air samples. The cassettes must be tightly sealed and packed in a material free of fibers or dust to minimize the potential for contamination. Plastic "bubble pack" is probably the most appropriate material for this purpose.

10. Sample Preparation

10.1 Under a negative flow HEPA hood (7.5), carefully wet-wipe the exterior of the cassettes to remove any possible contamination before taking cassettes into a clean preparation area.

10.2 Perform sample preparation in a clean facility that has a separate work area from both the bulk and air sample preparation areas.

10.3 Initial specimen preparation shall take place in a clean HEPA filtered negative pressure hood to avoid any possible contamination of the laboratory or personnel, or both, by the potentially large number of asbestos structures in an asbestos-containing dust sample. Cleanliness of the preparation area hoods is measured by the cumulative process blank surface loadings (see Section 11).

10.4 All sample preparation steps 10.4.1-10.4.6 shall take place in the dust preparation area inside a HEPA hood.

10.4.1 Remove the upper plug from the sample cassette and carefully introduce approximately 10 mL solution of a 50/50 mixture of particle-free water and reagent alcohol into the cassette using a plastic wash bottle (7.44). If the plugged nozzle was left attached to the cassette, then remove the plug and introduce the water/alcohol solution into the cassette through the tubing, and then remove the tubing, if it is visibly clean.

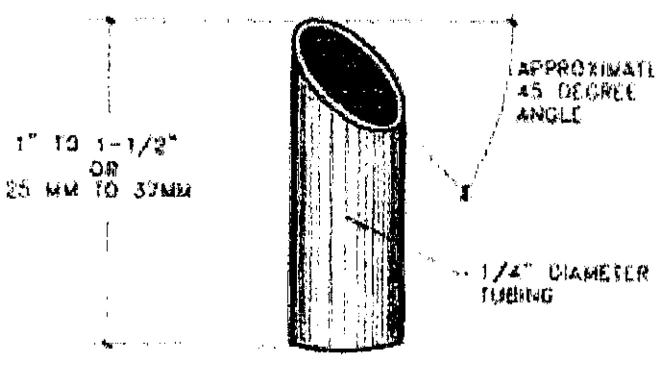

FIG. 1 Example of the Tubing Nozzle

10.4.2 Replace the upper plug or the sample cap and lightly shake the dust solution by hand for 3 s.

10.4.3 Remove the entire cap of the cassette and pour the suspension through a 1.0 by 1.0 mm opening screen (7.46) into a pre-cleaned 200 mL glass specimen bottle (7.9). All visible traces of the sample contained in the cassette shall be rinsed through the screen into the specimen bottle with a plastic wash bottle containing the 50/50 solution of particle-free water and alcohol. Repeat this procedure two additional times for a total of three washings. Next, rinse the nozzle two or three times through the screen into the specimen bottle with the 50/50 mixture of water and alcohol. Typically, the total amount of the 50/50 mixture used in the rinse is 50 to 75 mL. Discard the 1.0 by 1.0 mm screen and bring the volume of suspension in the specimen bottle up to the 100 mL mark on the side of the bottle with particle-free water only.

10.4.4 Adjust the pH of the suspension to 3 to 4 using a 10.0 % solution of acetic acid. Use pH paper for testing. Filter the suspension within 24 h to avoid problems associated with bacterial and fungal growth.

10.4.5 Use either a disposable plastic filtration unit or a glass filtering unit (7.14) for filtration of aliquots of the suspension. The ability of an individual filtration unit to produce a uniform distribution may be tested by the filtration of a colored particulate solution such as diluted India ink (solution of carbon black).

10.4.5.1 If a disposable plastic filtration unit is used, then unwrap a new disposable plastic filter funnel unit (either 25 or 47 mm diameter) and remove the tape around the base of the funnel. Remove the funnel and discard the top filter supplied with the apparatus, retaining the coarse polypropylene support pad in place. Assemble the unit with the adapter and a properly sized neoprene stopper, and attach the funnel to the 1000 mL side-arm vacuum flask (7.15). Place a 5.0 μm pore size MCE (backing filter) on the support pad. Wet it with a few mL of particle-free water and place an MCE (7.16) or PC filter (≤0.22 μm pore size) (7.17) on top of the backing filter. Apply a vacuum (7.36), ensuring that the filters are centered and pulled flat without air bubbles. Any irregularities on the filter surface requires the discard of that filter. After the filter has been seated properly, replace the funnel and reseal it with the tape. Return the flask to atmospheric pressure.

10.4.5.2 If a glass filtration unit is used, place a 5 μm pore size MCE (backing filter) on the glass frit surface. Wet the filter with particle-free water, and place an MCE or PC filter (≤0.22 μm pore size) on top of the backing filter. Apply a vacuum, ensuring that the filters are centered and pulled flat without air bubbles. Replace the filters if any irregularities are seen on the filter surface. Before filtration of each set of sample aliquots, prepare a blank filter by filtration of 50 mL of particle-free water. If aliquots of the same sample are filtered in order of increasing surface loading, the glass filtration unit need not be washed between filtration. After completion of the filtration, do not allow the filtration funnel assembly to dry because contamination is then more difficult to remove. Wash any residual solution from the filtration assembly by holding it under a flow of water, then rub the surface with a clean paper towel soaked in a detergent solution. Repeat the cleaning operation, and then rinse two times in particle-free water.

10.4.6 With the flask at atmospheric pressure, add 20 mL of particle-free water into the funnel. Cover the filter funnel with its plastic cover if the disposable filtering unit is used.

10.4.7 Briefly hand shake (3 s) the capped bottle with the sample suspension, then place it in a tabletop ultrasonic bath (7.12) and sonicate for 3.0 min. Maintain the water level in the sonicator at the same height as the suspension in sample bottle. The ultrasonic bath shall be calibrated as described in 20.5. The ultrasonic bath must be operated at equilibrium temperature. After sonicating, return the sample bottle to the work surface of the HEPA hood. Preparation steps 10.4.8-10.4.14 shall be carried out in this hood.

10.4.8 Shake the suspension lightly by hand for 3 s, then let it rest for 2.0 min to allow large particles to settle to the bottom of the bottle or float to the surface.

10.4.9 Estimate the amount of liquid to be withdrawn to produce an adequate filter preparation. Experience has shown that a light staining of the filter surface will yield a suitable preparation for analysis. Filter at least 1.0 mL, but no more than half the total volume. If after examination in the TEM, the smallest volume measured (1.0 mL) (7.13) yields an overloaded sample, then perform additional serial dilutions of the suspension. If it is estimated that less than 1.0 mL of suspension has to be filtered because of the density of the suspension, perform a serial dilution.

10.4.9.1 If serial dilutions are required, repeat step 10.4.8 before the serial dilution portion is taken. Do not re-sonicate the original suspension or any serial dilutions. The recommended procedure for a serial dilution is to mix 10 mL of the sample suspension with 90 mL of particle-free water in a clean sample bottle to obtain a 1:10 serial dilution. Follow good laboratory practices when performing dilutions.

10.4.10 Insert a new disposable pipette halfway into the sample suspension and withdraw a portion. Avoid pipetting any of the large floating or settled particles. Uncover the filter funnel and dispense the mixture from the pipette into the water in the funnel.

10.4.11 Apply vacuum to the flask and draw the mixture through the filter.

10.4.12 Discard the pipette.

10.4.13 Disassemble the filtering unit and carefully remove the sample filter with fine tweezers (7.11). Place the completed sample filter particle side up, into a precleaned, labeled, disposable, plastic petri dish (7.48) or other similar container.

10.4.14 In order to ensure that an optimally-loaded filter is obtained, it is recommended that filters be prepared from several different aliquots of the dust suspension. For this series of filters, it is recommended that the volume of each aliquot of the original suspension be a factor of five higher than the previous one. If the filters are prepared in order of increasing aliquot volume, all of the filters for one sample can be prepared using one plastic disposable filtration unit, or without cleaning of glass filtration equipment between individual filtration. Before withdrawal of each aliquot from the sample, shake the suspension without additional sonification and allow to rest for 2 min.

10.4.15 There are many practical methods for drying MCE filters. The following are two examples that can be used: (*1*) dry MCE filters for at least 12 h (over desiccant) in an airtight cabinet-type desiccator (7.21); (*2*) to shorten the drying time (if desired), remove a plug of the damp filter and attach it to a glass slide (7.19) as described in 12.1.2 and 12.1.3. Place the slide with a filter plug or filter plugs (up to eight plugs can be attached to one slide) on a bed of desiccant, in the desiccator for 1 h.

10.4.16 PC filters do not require lengthy drying before preparation, but shall be placed in a desiccator for at least 30 min before preparation.

10.5 Prepare TEM specimens from small sections of each dried filter using the appropriate direct transfer preparation method.

11. Blanks

11.1 Prepare sample blanks that include both a process blank (50 mL of particle-free water) for each set of samples analyzed and one unused filter from each new box of sample filters (MCE or PC) used in the laboratory. If glass filtering units are used, prepare and analyze a process blank each time the filtering unit is cleaned. Blanks will be considered contaminated, if after analysis, they are shown to contain more than 53 asbestos structures per square millimetre. This generally corresponds to three or four asbestos structures found in ten grid openings. The source of the contamination must be found before any further analysis can be performed. Reject samples that were processed along with the contaminated blanks and prepare new samples after the source of the contamination is found.

11.2 Prepare field blanks which are included with sample sets in the same manner as the samples, to test for contamination during the sampling, shipping, handling, and preparation steps of the method.

12. TEM Specimen Preparation of Mixed Cellulose Ester (MCE) Filters

NOTE 1—Use of either the acetone or the diamethylformamide-acetic acid method is acceptable.

12.1 *Acetone Fusing Method:*

12.1.1 Remove a section (a plug) from any quadrant of the sample and blank filters. Sections can be removed from the filters using a 7 mm cork borer (7.32). The cork borer must be wet wiped after each time a section is removed.

12.1.2 Place the filter section (particle side up) on a clean microscope slide. Affix the filter section to the slide with a gummed page reinforcement (7.43), or other suitable means. Label the slide with a glass scribing tool or permanent marker (7.10).

12.1.3 Prepare a fusing dish from a glass petri dish (7.37) and a metal screen bridge (7.38) with a pad of five to six ashless paper filters (7.42) and place in the bottom of the petri dish (4). Place the screen bridge on top of the pad and saturate the filter pads with acetone. Place the slide on top of the bridge in the petri dish and cover the dish. Wait approximately 5 min for the sample filter to fuse and clear.

12.2 *Dimethylformamide-Acetic Acid Method:*

12.2.1 Place a drop of clearing solution that consists of 35 % dimethylformamide (DMF), 15 % glacial acetic acid, and 50 % Type II water (v/v) on a clean microscope slide. Gauge the amount used so that the clearing solution just saturates the filter section.

12.2.2 Carefully lay the filter segment, sample surface upward, on top of the solution. Bring the filter and solution together at an angle of about 20° to help exclude air bubbles. Remove any excess clearing solution. Place the slide in an oven or on a hot plate, in a fume hood, at 65 to 70°C for 10 min.

12.3 Plasma etching of the collapsed filter is required.

12.3.1 The microscope slide to which the collapsed filter pieces are attached is placed in a plasma asher (7.27). Because plasma ashers vary greatly in their performance, both from unit to unit and between different positions in the asher chamber, it is difficult to specify the exact conditions that must be used. Insufficient etching will result in a failure to expose embedded fibers, and too much etching may result in the loss of particles from the filter surface. To determine the optimum time for ashing, place an unused 25 mm diameter MCE filter in the center of a glass microscope slide. Position the slide approximately in the center of the asher chamber. Close the chamber and evacuate to a pressure of approximately 40 Pa, while admitting oxygen to the chamber at a rate of 8 to 20 cm^3/min. Adjust the tuning of the system so that the intensity of the plasma is maximized. Determine the time required for complete oxidation of the filter. Adjust the system parameters to achieve complete oxidation of the filter in a period of approximately 15 min. For etching of collapsed filters, use these operating parameters for a period of 8 min. For additional information on calibration, see the *USEPA Asbestos-Containing Materials in Schools* (4) or *NIST/NVLAP Program Handbook for Airborne Asbestos Analysis* (6) documents.

12.3.2 Place the glass slide containing the collapsed filters into the low-temperature plasma asher, and etch the filter.

12.4 Carbon coating of the collapsed and etched filters is required.

12.4.1 Carbon coating must be performed with a high-vacuum coating unit (7.4), capable of less than 10^{-4} torr (13 MPa) pressure. Units that are based on evaporation of carbon filaments in a vacuum generated only by an oil rotary pump have not been evaluated for this application and shall not be used. Carbon rods (7.40) used for evaporators shall be sharpened with a carbon rod sharpener to a neck of about 4 mm in length and 1 mm in diameter. The rods are installed in the evaporator in such a manner that the points are approximately 100 to 120 mm from the surface of the microscope slide held in the rotating device.

12.4.2 Place the glass slide holding the filters on the rotation device, and evacuate the evaporator chamber to a vacuum of at least 13 MPa. Perform the evaporation in very short bursts, separated by 3 to 4 s to allow the electrodes to cool. An alternate method of evaporation is by using a slow continuous applied current. An experienced analyst can judge the thickness of the carbon film to be applied. Conduct tests on unused filters first. If the carbon film is too thin, large particles will be lost from the TEM specimen, and there will be few complete and undamaged grid openings on the specimen.

12.4.2.1 If the coating is too thick, it will lead to a TEM image that is lacking in contrast, and the ability to obtain electron diffraction patterns will be compromised. The carbon film shall be as thin as possible and still remain intact on most of the grid openings of the TEM specimen.

12.5 *Preparation of the Jaffe Washer*— The precise design of the Jaffe washer is not considered important, so any one of the published designs may be used (7, 8). One such washer consists of a simple stainless steel bridge contained in a glass petri dish.

12.5.1 Place several pieces of lens tissue (7.41) on the stainless steel bridge. The pieces of lens tissue shall be large enough to completely drape over the bridge and into the solvent. In a fume hood, fill the petri dish with acetone (or DMF) until the height of the solvent is brought up to contact the underside of the metal bridge as illustrated in Fig. 2.

12.6 *Placing the Specimens into the Jaffe Washer*:

12.6.1 Place the TEM grids (7.39) shiny side up on a piece of lens tissue or filter paper so that individual grids can be easily picked up with tweezers.

12.6.2 Prepare three grids from each sample.

12.6.2.1 Using a curved scalpel blade (7.20), excise at least two square (3 mm by 3 mm) pieces of the carbon-coated MCE filter from the glass slide.

12.6.2.2 Place the square filter piece carbon-side up on top of a TEM specimen grid.

12.6.2.3 Place the whole assembly (filter/grid) on the saturated lens tissue in the Jaffe washer.

12.6.2.4 Place the three TEM grid sample filter preparations on the same piece of lens tissue in the Jaffe washer.

12.6.2.5 Place the lid on the Jaffe washer and allow the system to stand for several hours.

12.7 Alternately, place the grids on a low level (petri dish filled to the ⅛ mark) DMF Jaffe washer for 60 min. Add enough solution of equal parts DMF/acetone to fill the washer to the screen level. Remove the grids after 30 min if they have cleared, that is, all filter material has been removed from the carbon film, as determined by inspection in the TEM.

12.8 Carefully remove the grids from the Jaffe washer, allowing the grids to dry before placing them in a clean marked grid box.

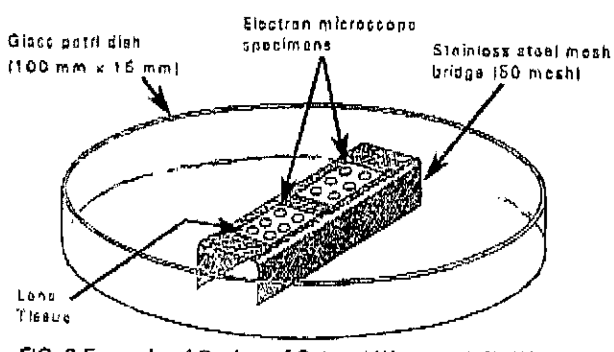

FIG. 2 Example of Design of Solvent Washer (Jaffe Washer)

13. TEM Specimen Preparation of Polycarbonate (PC) Filter

13.1 Cover the surface of a clean microscope slide with two strips of double-sided adhesive tape.

13.2 Cut a strip of filter paper slightly narrower than the width of the slide. Position the filter paper strip on the center of the length of the slide.

13.3 Using a clean, curved scalpel blade, cut a strip of the PC filter approximately 25 by 6 mm. Use a rocking motion of the scalpel blade to avoid tearing the filter. Place the PC strip particle side up on the slide perpendicular to the long axis of the slide. The ends of the PC strip must contact the double sided adhesive tape. Each slide can hold several PC strips. With a glass marker, label each PC strip with the individual sample number.

13.4 Carbon coat the PC filter strips as discussed in 12.4.2. PC filters do not require etching.

NOTE 2—**Caution:** Do not overheat the filter sections while carbon coating.

13.5 Prepare a Jaffe washer as described in 12.5, but fill the washer with chloroform or 1-methyl-2-pyrrolidone to the level of the screen.

13.6 Using a clean curved scalpel blade, excise three, 3-mm square filter pieces from each PC strip. Place the filter squares carbon side up on the shiny side of a TEM grid. Pick up the grid and filter section together and place them on the lens tissue in the Jaffe washer.

13.7 Place the lid on the Jaffe washer and rest the grids in place for at least 4 h. Best results are obtained with longer wicking times, up to 12 h.

13.8 Carefully remove the grids from the Jaffe washer, allowing the grids to dry before placing them in a clean, marked grid box.

14. Grid Opening Measurements

14.1 TEM grids must have a known grid opening area. Determine this area as follows:

14.2 Measure at least 20 grid openings in each of 20 random 75 to 100 µm (200-mesh) copper grids for a total of 400 grid openings for every 1000 grids used, by placing the 20 grids on a glass slide and examining them under the optical microscope. Use a calibrated graticule to measure the average length and width of the 20 openings from each of the individual grids. From the accumulated data, calculate the average grid opening area of the 400 openings.

14.3 Grid area measurements can also be made at the TEM at a calibrated screen magnification of between 15 000 and 20 000X. Typically measure one grid opening for each grid examined. Measure grid openings in both the x and y directions and calculate the area.

14.4 Pre-calibrated TEM grids are also acceptable for this test method.

15. TEM Method

15.1 Microscope settings: 80 to 120 kV, 15 000 to 20 000X screen magnification for analysis (7.2).

15.2 Analyze two grids for each sample. Analyze one-half of the sample area on one sample grid preparation and the remaining half on a second sample grid preparation.

15.3 *Determination of Specimen Suitability:*

15.3.1 Carefully load the TEM grid, carbon side facing up (in the TEM column) with the grid bars oriented parallel/perpendicular to the length of the specimen holder. Use a hand lens or loupe, if necessary. This procedure will line up the grid with the *x* and *y* translation directions of the microscope. Insert the specimen holder into the microscope.

15.3.2 Scan the entire grid at low magnification (250X to 1000X) to determine its suitability for high magnification analysis as specified in 15.3.3.

15.3.3 Grids are acceptable for analysis if the following conditions are met:

15.3.3.1 The fraction of grid openings covered by the replica section is at least 50 %.

15.3.3.2 Relative to that section of the grid covered by the carbon replica, the fraction of intact grid openings is greater than 50 %.

15.3.3.3 The fractional area of undissolved filter is less than 10 %.

15.3.3.4 The fraction of grid openings with overlapping or folded replica film is less than 50 %.

15.3.3.5 At least 20 grid openings, that have no overlapping or folded replica, are less than 5 % covered with holes and have less than 5 % opaque area due to incomplete filter dissolution.

15.4 *Determination of Grid Opening Suitability:*

15.4.1 If the grid meets acceptance criteria, choose a grid opening for analysis from various areas of the grid so that the entire grid is represented. Determine the suitability of each individual grid opening prior to the analysis.

15.4.2 The individual grid opening must have less than 5 % holes over its area.

15.4.3 Grid openings must be less than 25 % covered with particulate matter.

15.4.4 Grid openings must be uniformly loaded.

15.5 Observe and record the orientation of the grid at 80 to 150X, on a grid map record sheet along with the location of the grid openings that are examined for the analysis. If indexed grids are used, a grid map is not required, but the identifying coordinates of the grid square must be recorded.

16. Recording Data Rules

16.1 Record on the count sheet any continuous grouping of particles in which an asbestos fiber is detected. Classify asbestos structures as fibers, bundles, clusters, or matrices as defined in 5.2.

16.2 Use the criteria for fiber, bundle, cluster, and matrix identification, as described in the *USEPA Asbestos-Containing Materials in Schools* document (4). Record, for each AHERA structure identified, the length and width measurements.

16.3 Record NSD (No Structures Detected) when no structures are detected in the grid opening.

16.4 Identify structures classified as chrysotile identified by either electron diffraction or X-ray analysis (7.3) and recorded on a count sheet. Verify at least one out of every ten chrysotile structures by X-ray analysis.

16.5 Structures classified as amphiboles by X-ray analysis and electron diffraction are recorded on the count sheet. For more information on identification, see Yamate, et al, (7) or Chatfield and Dillon (8).

16.6 Record a typical electron diffraction pattern for each type of asbestos observed for each group of samples (or a minimum of every five samples) analyzed. Record the micrograph number on the count sheet. Record at least one X-ray spectrum for each type of asbestos observed per sample. Attach the print-outs to the back of the count sheet. If the X-ray spectrum is stored, record the file and disk number on the count sheet.

16.7 *Counting Rules:*

16.7.1 At a screen magnification of between 15 000 and 20 000X evaluate the grids for the most concentrated sample loading; reject the sample if it is estimated to contain more than 50 asbestos structures per grid opening. Proceed to the next lower concentrated sample until a set of grids are obtained that have less than 30 asbestos structures per grid opening.

16.8 *Analytical Sensitivity (AS)*—As determined by the following equation:

$$(EFA \times 100\ mL \times 1)/(GO \times GOA \times V \times SPL) = AS \quad (1)$$

where:

EFA = effective filter area of the final sampling filter, mm^2
GO = number of grid openings counted
GOA = average grid opening area, mm^2
SPL = surface area sampled, cm^2
V = volume of sample filtered in step 10.4.9, representing the actual volume taken from the original 100 mL suspension, mL
AS = analytical sensitivity, expressed as asbestos structures/cm^2

16.8.1 An AS of approximately 1000 asbestos structures per square centimetre (calculated for the detection of a single asbestos structure) has been designed for this analysis. This sensitivity can be achieved by increasing the amount of liquid filtered, increasing the number of grid openings analyzed, increasing the area of the collection, or decreasing the size of the final filter. For example, using a collection area = 500 cm^2, filter area = 1000 mm^2, number of grid openings = 10, and a grid area of 0.01 mm^2, V = 50 mL, the AS is 40 str/cm^2. Occasionally, due to high particle loadings or high asbestos surface loading, this AS cannot be practically achieved and stopping rules apply.

16.8.2 The numerical value of AS required for any specific application of this method may be achieved by selecting an appropriate combination of the sampling and analysis parameters in the above equation. For example, if SPL = 100 cm^2, EFA = 1000 mm^2, GO = 10, GOA = 0.01 mm^2, V = 10 mL, and D = 1, then AS = 1000 str/cm^2. Increasing GO to 50 and V to 50 mL changes the AS to 40 Str/cm^2.

16.8.3 When sample filters are heavily loaded with particulate matter, it may useful to employ serial dilutions during preparation to reduce the loading on grid specimens to acceptable levels and thus facilitate analysis. Under such circumstances, the AS may be calculated by substituting an appropriate value for the dilution factor, D, into the above equation. In general:

$$D = VA/(V + VPFW) \quad (2)$$

VA = the volume of the aliquot from the new, diluted suspension that is filtered to prepare the analytical filter; V = the volume of the aliquot from the initial (100 mL) suspension that is diluted; and VPFW = the volume of particle free water added to V during serial dilution to produce the new, diluted suspension.

Thus, if GO = 10, V = 10 mL, VPFW = 90 mL, and VA = 1.0 mL, D = 0.01 and the AS = 100 000 str/cm^2.

16.9 *Limit of Detection*—The limit of detection for this test method is calculated using the Practice D 6620. All data shall be provided in the laboratory report.

16.10 *Stopping Rules*:

16.10.1 The analysis is stopped upon the completion of the grid square that achieves an AS of less than 1000 asbestos structures per square centimetre.

16.10.2 If an AS of 1000 asbestos structures per square centimetre cannot be achieved after analyzing ten grid openings then stop on grid opening No. 10 or the grid opening which contains the 100th asbestos structure, whichever comes first. A minimum of four grid squares shall be analyzed for each sample.

16.10.2.1 If the analysis is stopped because of the 100th structure rule, the entire grid square containing the 100th structure must be counted.

16.11 After analysis, remove the grids from the TEM, and replace them in the appropriate grid storage holder.

17. Sample Storage

17.1 The washed-out sample cassettes can be discarded after use.

17.2 Sample grids and unused filter sections (7.18) must be stored for a minimum of one year.

18. Reporting

18.1 Report the following information for each dust sample analyzed:

18.1.1 Surface loading in structures/cm^2.

18.1.2 The AS.

18.1.3 Types of asbestos present.

18.1.4 Number of asbestos structures counted.

18.1.5 Effective filtration area.

18.1.6 Average size of the TEM grid openings that were counted.

18.1.7 Number of grid openings examined.

18.1.8 Sample dilution used.

18.1.9 Area of the surface sampled.

18.1.10 Listing of size data for each structure counted.

18.1.11 A copy of the TEM count sheet or a complete listing of the raw data. An example of a typical count sheet is shown in Appendix X1.

18.2 Determine the amount of asbestos in any accepted sample using the following formula:

$$\frac{EFA \times 100 \text{ mL} \times \#STR}{GO \times GOA \times V \times SPL} = \text{asbestos structures/cm}^2 \quad (3)$$

where:

$\#STR$ = number of asbestos structures counted,
EFA = effective filter area of the final sampling filter, mm^2,
GO = number of grid openings counted,
GOA = average grid opening area, mm^2,
SPL = surface area sampled, cm^2, and
V = volume of sample filtered in step 10.4.9, representing the actual volume taken from the original 100 mL suspension, mL.

19. Quality Control/Quality Assurance

19.1 In general, the laboratory's quality control checks are used to verify that a system is performing according to specifications regarding accuracy and consistency. In an analytical laboratory, spiked or known quantitative samples are normally used. However, due to the difficulties in preparing known quantitative asbestos samples, routine quality control testing focuses on re-analysis of samples (duplicate recounts).

19.1.1 Re-analyze samples at a rate of 1/10 of the sample sets (one out of every ten samples analyzed not including laboratory blanks). The re-analysis shall consist of a second sample preparation obtained from the final filter.

19.2 In addition, quality assurance programs must follow the criteria shown in the *USEPA Asbestos-Containing Materials in Schools* document (4) and in the *NIST/NVLAP Program Handbook for Airborne Asbestos Analysis* document (6). These documents describe sample custody, sample preparation, blank checks for contamination, calibration, sample analysis, analyst qualifications, and technical facilities.

20. Calibrations

20.1 Perform calibrations of the instrumentation on a regular basis, and retain these records in the laboratory, in accordance with the laboratory's quality assurance program.

20.2 Record calibrations in a log book along with dates of calibration and the attached backup documentation.

20.3 A calibration list for the instrument is as follows:

20.3.1 *TEM*:

20.3.1.1 Check the alignment and the systems operation. Refer to the TEM manufacturer's operational manual for detailed instructions.

20.3.1.2 Calibrate the camera length of the TEM in electron diffraction (ED) operating mode before ED patterns of unknown samples are observed. Camera length can be measured by using a carbon coated grid on which a thin film of gold has been sputtered or evaporated. A thin film of gold is evaporated on the specimen TEM grid to obtain zone-axis ED patterns superimposed with a ring pattern from the polycrystalline gold film. In practice, it is desirable to optimize the thickness of the gold film so that only one or two sharp rings are obtained on the superimposed ED pattern. Thick gold films will tend to mask weak diffraction spots from the fibrous particles. Since the unknown d-spacings of most interest in asbestos analysis are those which lie closest to the transmitted beam, multiple gold rings from thick films are unnecessary. Alternatively, a gold standard specimen can be used to obtain an average camera constant calculated for that particular instrument and can then be used for ED patterns of unknowns taken during the corresponding period.

20.3.1.3 Perform magnification calibration at the fluorescent screen. This calibration must be performed at the magnification used for structure counting. Calibration is performed with a grating replica (7.47) (for example, one containing at least 2160 lines/mm).

(a) Define a field of view on the fluorescent screen. The field of view must be measurable or previously inscribed with a scale or concentric circles (all scales should be metric).

(b) Frequency of calibration will depend on the service history of the particular microscope.

(c) Check the calibration after any maintenance of the microscope that involves adjustment of the power supply to the lens or the high voltage system or the mechanical disassembly of the electron optical column (apart from filament exchange).

(d) The analyst must ensure that the grating replica is placed at the same distance from the objective lens as the specimen.

(e) For instruments that incorporate a eucentric tilting specimen stage, all specimens and the grating replica must be placed at the eucentric position.

20.3.1.4 The smallest spot size of the TEM must be checked.

(a) At the crossover point, photograph the spot size at a screen magnification of 15 000 to 20 000X. An exposure time of 1 s is usually adequate.

(b) The measured spot size must be less than or equal to 250 nm.

20.4 *EDXA*:

20.4.1 The resolution and calibration of the EDXA must be verified.

20.4.1.1 Collect a standard EDXA Cu peak from the Cu grid.

20.4.1.2 Compare the X-ray energy versus channel number for the Cu peak and be certain that readings are within ±10 eV.

20.4.2 Collect a standard EDXA of crocidolite asbestos (NIST SRM 1866).

20.4.2.1 The elemental analysis of the crocidolite must resolve the Na peak.

20.4.3 Collect a standard EDXA of chrysotile asbestos.

20.4.3.1 The elemental analysis of chrysotile must resolve both Si and Mg on a single chrysotile fiber.

20.5 Ultrasonic bath calibration shall be performed as follows:

20.5.1 Fill the bath water to a level equal to the height of suspension in the glass sample container that will be used for the dust analysis. Operate the bath until the water reaches the equilibrium temperature.

20.5.2 Place 100 mL of water (at approximately 20°C) in another 200-mL glass sample container, and record its temperature.

20.5.3 Place the sample container in the water in the ultrasonic bath (with the power turned off). After 60 s, remove the glass container and record its temperature.

20.5.4 Place 100 mL of water (at approximately 20°C) in another 200-mL glass sample container, and record its temperature.

20.5.5 Place the second sample container into the water in the ultrasonic bath (with the power turned on). After 60 s, remove the glass container and record its temperature.

20.5.6 Calculate the rate of energy deposition into the sample container using the following formula:

$$R = 4.185 \times \sigma \times \rho \times \frac{(\theta_2 - \theta_1)}{t} \tag{4}$$

where:

4.185	=	Joules/cal,
R	=	energy deposition, watts/mL,
θ_1	=	temperature rise with the ultrasonic bath not operating, °C,
θ_2	=	temperature rise with the ultrasonic bath operating, °C,
t	=	time in seconds, 60 s (20.5.3 and 20.5.5),
σ	=	specific heat of the liquid in the glass sample container, 1.0 cal/g, and
ρ	=	density of the liquid in the glass sample container, 1.0 g/cm^3.

20.5.7 Adjust the operating conditions of the bath so that the rate of energy deposition is in the range of 0.08 to 0.12 MW/m 3, as defined by this procedure.

21. Precision and Bias

21.1 *Precision*—The precision of the procedure in this test method is being determined using round robin data from participating laboratories.

21.2 *Bias*—Since there is no accepted reference material suitable for determining the bias of the procedure in this test method, bias has not been determined (see Specification D 3670).

NOTE 3—Round robin data is under development and will be presented as a research report.

22. Keywords

22.1 asbestos; microvacuuming; settled dust; TEM

APPENDIX

(Nonmandatory Information)

X1. DUST SAMPLE ANALYSIS

X1.1 See Figs. X1.1 and X1.2 for the dust analysis worksheet and the TEM count sheet.

DUST SAMPLE ANALYSIS

Client:		Accelerating Voltage:	
Sample ID:		Indicated Mag:	KX
Job Number:		Screen Mag:	KX
Date Sample Analyzed:	- -	Microscope:	1 2 3 4 5
Number of Openings/Grids Counted:		Filter Type:	
Grid Accepted, 600X:	Yes No	Filter Size:	
Percent Loading:	%	Filter Pore Size (μm):	
Grid Box #1:		Grid Opening:	1) μm x μm
			2) μm x μm

Analyst:

Reviewer:

Counting Rules: AHERA LEVEL II

Calculation Data:

Effective Filter Area in mm^2:	(EFA)	
Number of Grid Openings Counted:	(GO)	
Average Grid Opening Area in mm^2:	(GOA)	
Volume of sample Filtered in ml:	(V)	
Surface area Sampled in cm^2:	(SPL)	
Number of Asbestos Structures Counted:*	(#STR)	

* If the number of asbestos structures counted is less than or equal to 4, enter 4 structures as the limit of detection here.

FORMULA FOR CALCULATION OF ASBESTOS STRUCTURES "DUST" PER CM^2:

$$\frac{EFA X \; 100 \; X \; \#STR}{GO \; X \; GOA \; X \; VX \; SPL} = \text{(Asbestos Structures per cm}^2\text{)}$$

Results for Total Asbestos Structures: ______ (Structures per cm^2)

Results for Structures ≥ microns: ______ (Structures per cm^2)

FIG. X1.1 Dust Sample Analysis Work Sheet

Job Number: ____________________

Structure #	Grid # Square #	Type	Structure	Length Microns	Width Microns	Confirmation		
						Morph.	SAED	EDS

Note: Keys to Abbreviations Used in Figure:

Type:

C = Chrysotile
AM = Amosite
CR = Crocidolite
AC = Actinolite
TR = Tremolite
AN = Anthophyllite
N = Non Asbestos

Structure:

F = Fiber
B = Bundle
C = Cluster
M = Matrix

Others:

NSD = No Structures Detected
Morph = Morphology
SAED = Selected Area Electron Diffraction
EDS = Energy Dispersive X-Ray Spectroscopy
ER = Inter-Row Spacing
NP = No Pattern

FIG. X1.2 TEM Count Sheet

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